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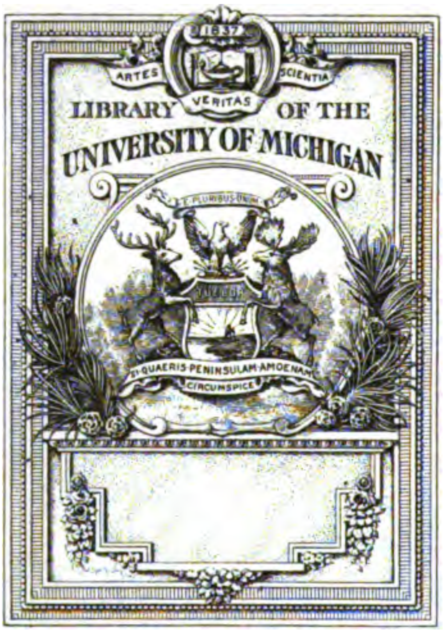

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# PRACTICAL ASSAYING.

LONDON: PRINTED BY
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# A MANUAL

OF

# PRACTICAL ASSAYING.

BY

JOHN MITCHELL, F.C.S.

THIRD EDITION

EDITED BY

WILLIAM CROOKES, F.R.S.

ETC.

LONDON:
LONGMANS, GREEN, AND CO.
1868.

# PREFACE

TO

# THE THIRD EDITION.

05-28-36 MLcu

THE RAPID PROGRESS of Science renders a metallurgical work antiquated in a comparatively short period, so that the last (second) edition of the late Mr. Mitchell's 'Manual of Practical Assaying,' published in 1854, is no longer in accordance with our enlarged knowledge of the subject.

In this edition are incorporated all the late important discoveries in Assaying made in this country and abroad, and special care is devoted to the very important Volumetric and Colorimetric Assays, as well as to the Blowpipe Assays. Most of the chapters are entirely re-written, whilst the chapter on Crystallography—being a subject only remotely bearing on Assaying — is left out altogether. On the other hand, in some cases, it may seem that by treating of purely analytical details the limits of Assaying have been exceeded. But these departments are so closely related as to make it impossible to fix the line of demarcation between them. Moreover, chemistry is cultivated by almost all to whom this work is of interest or service, so that it is hoped these amplifications will add to its value.

The old equivalents are retained, as they are more generally understood by students of science who do not make chemistry their chief study.

The Editor is under many obligations to his friend Dr. Röhrig, M.E., for assistance in revising the manuscript and incorporating into the work the latest continental improvements, as set forth in Professor Kerl's *Probirkunst.* The author of the best work on volumetric analysis which has yet appeared in English, Mr. Sutton, F.C.S., has kindly placed several cuts, &c., at the Editor's disposal, and some descriptions of German processes have been taken from the last English Edition of Fresenius's *Quantitative Analysis*—a work which should be a standard of reference for all students who desire to carry their chemical researches further than is possible to be treated of in a work professing to deal only with Assaying.

LONDON: *September*, 1868.

# PREFACE

TO

# THE SECOND EDITION.

IN PRESENTING this the Second Edition of the 'Manual of Practical Assaying' to his mining friends and the public in general, the author has to tender his sincere thanks for the very favourable opinion expressed of the former edition, which was honoured with a most extensive circulation, not only in the United Kingdom, but in all the Colonies, the United States, and South America: in addition to which it was translated into Spanish, for the use of the Government School of Mines at Madrid.

The former edition having been out of print for some time, repeated calls have been made on the author for a Second Edition; and, in compliance with this general request, the present volume has been prepared. The same arrangement (as far as practicable) has been adhered to as in the first edition, but a considerable portion has been entirely re-written, and much new matter added. It is also embellished with nearly 400 engravings illustrative of crystallography, and the various apparatus described in the body of the work.

In its preparation, the author has been greatly influenced by a desire to extend the sphere of utility of the former edition, by introducing, in the smallest possible space, and in the simplest form, such instructions in elementary chemistry, chemical notation, the use of chemical symbols, &c. as will enable the assayer or metallurgist to trace the varied re-actions occurring either in the crucible or the furnace during the progress of an experiment.

Crystallography has also been made the subject of attention with a view to the discrimination of mineral substances by crystalline forms, aided by a few chemical tests.

Under the assay of Silver, there is added a full and complete description of the mode of assay employed in the Paris Mint, together with engravings of the apparatus in use.

A chapter has also been introduced, containing full instructions for the discrimination of all the more commonly occurring Gems and Precious Stones; and in the Appendix will be found copious Tables for the Valuation of Gold of every degree of fineness, expressed either in carats or thousandths; following which is an Assay Table, for calculating the number of ounces, pennyweights, and grains of gold or silver in a ton of mineral, when a given quantity has been submitted to assay.

In conclusion, the astounding discoveries of mineral wealth which are now daily being made, not only in this country, but in every other to which a due amount of diligence and information has been turned, renders the appearance of a complete Manual of the more closely allied branches of knowledge involved in the successful cultivation of such researches a desideratum of considerable importance.

The present volume, it is hoped, will fill the existing void in the literature subservient to this branch of our knowledge.

Assay Office,
Dunning's Alley, Bishopsgate Street Without.

# PREFACE

TO

# THE FIRST EDITION.

WHEN the rank our country holds among nations, as regards her Mining interest, is taken into consideration, it must be with all a matter of surprise that no work especially devoted to the elucidation of the processes to be employed in ascertaining the richness in metal of any sample of ore (that is, in other terms, its Assay) has of late years appeared before the British public. Indeed, the only work at present known in England is Berthier's 'Traité des Essais par la Voie Sèche,' which, for the mere purpose of inculcating the principles of assaying, has many disadvantages—not the least of which is its being written in a foreign tongue; and although a knowledge of French is now so very general yet many are prevented buying scientific works in that language on account of the difficulties of finding equivalents for the technicalities which must necessarily be employed. It is also a very large work, and one containing much matter which the assayer does not need—matter relating to the composition of wood and coal ashes, furnace products, &c. which are more especially adapted for the metallurgist.

These considerations, coupled with the paucity of any knowledge of assaying, excepting that confined to a very limited number of persons, induced the author of the following pages to turn a considerable amount of his attention to this subject; more especially as much difficulty was experienced in not having a suitable text-book for the use of his pupils. A portion of the following pages was drawn up as a Manual for such a purpose; but on consideration, it was thought the extension of such a work was so much needed that it was determined to alter the original plan as far as was consistent with the complete carrying out of the object in view, viz. the production of a Manual embodying information in every branch of assaying, either by the wet or the dry processes.

The following is a sketch of the manner in which this is accomplished; the author having followed the excellent arrangement of Berthier as closely as possible, from whose work also much matter that suited these pages, and which it would have been useless to re-write, has been inserted. Firstly, the Mechanical and Chemical Operations of Assaying are treated in full, inclusive of a description of the apparatus required, their mode of use, &c. Secondly, Furnaces, Fuel, and Crucibles, together with a description of the best Pyrometers, and their applications. Thirdly, the Fluxes, their properties, preparation, use, &c. Fourthly, an Essay on the use of the Blowpipe, and all its appurtenances; as Fluxes, Supports, &c. Fifthly, the action of the Fluxes on some Mineral Substances. Sixthly, a method of discriminating many Minerals by means of the Blowpipe, aided by a few tests by the humid method. Seventhly, the Humid Analysis of many Mineral Substances, their composition, locality, &c. (All the minerals mentioned in the three last heads comprehend such only as generally come under the notice of the Assayer.) Eighthly, the complete Assay of all the common Metals, in addition to which the Assay of Sulphur, Chromium, Arsenic, Heating power of Fuel, &c. is fully discussed; and ninthly, and lastly, a copious Table drawn up for the purpose of ascertaining in Assays of Gold and Silver the precise amount, in ounces, pennyweights, and grains, of Noble metal contained in a Ton of Ore from the assay of a given quantity. This Table is the most complete and copious yet published.

Not only has it been endeavoured to collect all that is generally known on the subject of Assaying, but many new facts have been added, and such matter entered into, that the success of an assay is rendered much more certain; and most assays are conducted more rapidly and with greater exactitude than heretofore.

It has also been endeavoured to introduce a new system, in which is pointed out the rationale of each process, with the chemical action taking place between the fluxes and the ores in course of assay, so that by paying a careful attention to the matters discussed, so much of the chemical nature of all ores that can come under the assayer's hand may be known, that the practice by 'rule of thumb' (a rule on which very little dependence is to be placed, excepting after years of the most laborious practice, and a rule which cannot be imparted, excepting the pupil pursue the same unprofitable course) must, it is hoped, be speedily abandoned when, by knowing the chemical properties of the body operated on, the necessary fluxes and processes might be at once indicated, and with a certainty of perfect success.

Having premised thus much, the author must beg to express his thanks to his friend Mr. F. Field for the kind assistance he afforded him whilst experimenting on the various modes of assay described in the body of the work; and trusting that any little imperfections which may be detected will not be harshly criticised, but that it may be taken into consideration that the author has attempted to improve a branch of mining knowledge to which unfortunately too little attention has been devoted, and to which, if he has added anything useful, he is indebted for the first principles of such knowledge to Berthier's 'Traité des Essais,' for which, to the talented writer of the above work, he is under the most lasting obligation.

23 Hawley Road, Kentish Town, London.

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# MANUAL
OF
# PRACTICAL ASSAYING.

## CHAPTER I.

### CHEMICAL NOMENCLATURE: LAWS OF COMBINATION: ETC.

It is not necessary, neither is it possible, in a treatise like the present, to give more than a very brief outline of the elements of chemical nomenclature, and the more simple laws of chemical combination. Undoubtedly a knowledge of chemistry is of the greatest value to the practical assayer —indeed, we may say no one can attain to any degree of eminence in this branch of industry unless he has had some amount of practice in the laboratory. But it will be quite beyond our province here to teach the elements of chemistry. So much of the nomenclature and leading laws will be given as are indispensable to a proper understanding of what is to follow, but beyond this the student must seek for further instruction in chemistry from books which are specially devoted to that science; and he should above all things endeavour to acquire a practical acquaintance with the laws and manipulations of experimental chemistry, by taking a series of lessons in a laboratory.

**Chemical Nomenclature.**—Every material substance with which we are acquainted consists of one or more bodies, termed *simple* or *elementary*—such bodies being so called from the fact that with our present means of research we are unable to reduce them to a more simple form. Thus, if a piece of common iron pyrites, or mundic, as it is commonly called, be submitted to certain operations, it will be found that we can obtain from it two substances totally distinct, in both physical and chemical properties, from each other and from the substance from which they were obtained. One body is *sulphur*, an opaque yellow substance, fusing at a very low temperature, igniting readily, and exhaling when burning a peculiar and suffocating odour. The other constituent of the pyrites is *iron*, a well-known metallic substance, requiring an intense heat for fusion, and becoming red-hot without burning. If we were now to perform any experiment which, in the present state of knowledge, ingenuity could suggest, we should be totally unable to cause either the sulphur or the iron to assume a more simple or elementary state of existence. We can with ease cause either of them to enter into new combinations with other bodies, and these compounds we can decompose—as in the case of the pyrites—and obtain both sulphur and iron again in their separate forms with all their characteristic properties; but nothing more than this can be effected: hence we are led to the belief that both sulphur and iron are simple bodies, or bodies containing only one kind of matter.

The following is a list of the simple substances discovered up to the present time; it is accompanied by certain symbols and numbers, the use and nature of which will be hereafter pointed out. Those substances in italics have hitherto found no practical use; and those marked with an asterisk (*) are often found native, or unassociated with mineralising elements.

The first column contains the common name of the element; the second, the symbol, or chemical short-hand character, in which all chemical changes and decompositions are most readily written and understood; and the third, the atomic weights. These atomic weights are not absolutely correct: they are not given beyond the first place of decimals, to avoid tedious calculation. For all practical purposes they may be considered accurate. Of the compounds of these elements, only those will be discussed which are likely to fall under the notice of the assayer.

*Non-Metallic Elements or Metalloids.*

| Names of the Elements | Symbols | Atomic Weights | Names of the Elements | Symbols | Atomic Weights |
|---|---|---|---|---|---|
| Oxygen . . | O | 8 | *Selenium* . . | Se | 39·7 |
| Hydrogen . . | H | 1 | *Tellurium* . . | Te | 64·5 |
| Nitrogen . . | N | 14 | Phosphorus . | P | 31 |
| Fluorine . . | Fl | 19 | Arsenic . . | As | 75 |
| Chlorine . . | Cl | 35·5 | *Carbon . . | C | 6 |
| Bromine . . | Br | 80 | Boron . . | B | 11 |
| Iodine . . | I | 127 | Silicium . . | Si | 14 |
| *Sulphur . . | S | 16 | *Zirconium* . . | Zr | 44·8 |

*Metallic Elements.*

| Names of the Elements | Symbols | Atomic Weights | Names of the Elements | Symbols | Atomic Weights |
|---|---|---|---|---|---|
| Potassium . . | K | 39·1 | Nickel . . | Ni | 29·5 |
| Sodium . . | Na | 23 | Zinc . . . | Zn | 32·6 |
| *Lithium* . . | Li | 7 | Cadmium . . | Cd | 56 |
| Barium . . | Ba | 68·5 | *Copper . . | Cu | 31·7 |
| Strontium . . | Sr | 43·8 | Lead . . . | Pb | 103·5 |
| Calcium . . | Ca | 20 | *Thallium* . . | Tl | 203 |
| Magnesium . | Mg | 12 | *Bismuth . . | Bi | 210 |
| Aluminium . | Al | 13·5 | Tin . . . | Sn | 59 |
| *Glucinum* . . | Be | 4·7 | Titanium . . | Ti | 25 |
| *Cerium* . . | Ce | 46 | *Tungsten* . . | W | 92 |
| *Lanthanum* . | La | 46 | *Molybdenum* . | Mo | 48 |
| *Didymium* . . | Di | 48 | *Vanadium* . . | V | 68·6 |
| *Yttrium* . . | Y |  | Antimony . . | Sb | 122 |
| *Erbium* . . | Er |  | *Mercury . . | Hg | 100 |
| *Terbium* . . | Tr |  | *Silver . . | Ag | 108 |
| *Niobium* . . | Nb |  | **Rhodium* . . | Rh | 52·2 |
| *Tantalum* . . | Ta | 37·6 | **Palladium* . . | Pd | 53·3 |
| *Thorium* . . | Th | 59·5 | *Platinum . . | Pt | 98·7 |
| Manganese . | Mn | 27·5 | **Iridium* . . | Ir | 99 |
| Chromium . | Cr | 26·7 | **Ruthenium* . | Ru | 52·2 |
| *Uranium* . . | U | 60 | **Osmium* . . | Os | 99·6 |
| Iron . . . | Fe | 28 | *Gold . . . | Au | 197 |
| Cobalt . . | Co | 29·5 |  |  |  |

The principal compound bodies are acids, oxides, salts, and binary substances containing no oxygen.

When a body combines in more than one proportion with oxygen, that compound containing the least oxygen takes the termination *ous*, that the most *ic*; thus, sulphurous acid, sulphuric acid; arsenious acid, arsenic acid; ferrous oxide, ferric oxide; mercurous oxide; in only one proportion (or when they form only one *basic* oxide) they are distinguished by the termination *ic*, as potassic oxide, aluminic oxide.

**Oxides** are binary oxygen compounds: they may be divided into two series. The first comprises those oxides which do not possess the property of combining with acids to form salts—they are termed *indifferent oxides*; the second series contains those capable of uniting with acids to form salts, and are called *salifiable oxides* or *bases*.

When a simple body, in combining with oxygen, forms but one oxide, it is simply called an *oxide*, added to the name of the simple body; thus, *oxide of zinc* or *zincic oxide.*

If the body is capable of combining with oxygen in many proportions, the words *proto*, *sesqui*, *bin* or *per*, &c., precede the term *oxide* to express the progressive amounts of oxygen. Most metals form one salifiable oxide, and many of them have two. These are now generally distinguished by the terminations *ous* and *ic*, in the same manner as are the acids; thus, we have *protoxide* of lead, iron, copper, tin, &c.; *sesquioxide* of aluminium, iron or chromium, &c.; *binoxide*, or *peroxide* of manganese, copper, mercury, &c.; and when we speak of them as salifiable bases, ferrous oxide and ferric oxide; mercurous and mercuric oxide; potassic oxide; aluminic oxide.

There are still higher degrees of oxidation of some metals; these are nearly always acids—as chromic, stannic, and antimonic acids.

**Salts** are formed when an acid unites with a base, and usually the properties of the acid and the base are reciprocally neutralised; thus, an acid which before combination possessed the power of reddening blue litmus, loses it in proportion as it combines with the base; and in like manner a base which would at first change reddened litmus paper to blue, loses this property as the acid saturates it. In this case the acid and base have combined to form a salt.

In naming salts, we have to consider—firstly, the nature of the acid; secondly, the salifiable nature of the base; and thirdly, the proportions in which the acid and base are combined.

Acids terminating in *ic* form salts terminating in *ate.*

Acids terminating in *ous* form salts terminating in *ite*; and the new names terminating in *ate* and *ite* are added to the name of the compound oxide. Thus, sulphuric acid and protoxide of iron form sulphate of protoxide of iron, ferrous sulphate, or, more commonly, protosulphate of iron. Arsenious acid and protoxide of iron form arsenite of protoxide of iron, ferrous arsenite, or protarsenite of iron; nitric acid and sesquioxide of iron, nitrate of sesquioxide of iron or ferric nitrate.

When the salt formed exists in the neutral state, its name is formed as above; but if the proportion of acid is larger than in neutral salts, it is termed an *acid salt*: thus we have *acid sulphate of potash.*

If the base is in excess, the name is preceded by *sub*: thus, *subacetate of lead.* This class of salt is also called *basic.*

**BINARY COMPOUNDS CONTAINING NO OXYGEN.**—These compounds exist very largely in nature, and it is from them we obtain the greater part of our copper, lead, silver, &c.

When a metalloid combines with a metal to form a compound which is neither acid nor basic, its name is derived from the metalloid by the addition of the termination *uret* or *ide.* The latter is more usually employed by chemists; the former by miners and mineralogists. The latter term is however gradually displacing the former, and is now seldom applied except to the sulphur compounds, and in these one term is as often used as the other; for the sake of uniformity, it is much to be desired that the termination *uret* should be discontinued altogether. Thus the compounds of sulphur and chlorine with iron and silver, are called *sulphuret* or *sulphide* of iron, *chloride* of silver, &c.

If a metalloid combines with a metal in more than one proportion, the same rule is followed as in the oxygen compounds; thus we have *proto*-sulphide of iron, *sesqui*-sulphide of iron, and *bi*-sulphide of iron (ordinary iron pyrites or mundic).

**LAWS OF COMBINATION.**—On examining the variety of compounds which the same substances may afford by their union in different proportions, it has been discovered that the proportions of the elements existing in each compound are definite; a certain weight of one substance will only combine with a certain weight or weights of another substance, and the lowest combining weight of any of the elementary bodies is termed its *equivalent* or *atomic weight*, and is represented by the numbers in the third column of the table of elementary substances.

As before stated, all substances combine in fixed or definite proportions; thus, if 111½ parts of oxide of lead were analysed, they will be found to consist of 103½ parts of lead and 8 of oxygen. Again, the analysis of 9 parts of water or oxide of hydrogen would give 1 part of hydrogen and 8 of oxygen; now, assuming, as in the table of equivalents, hydrogen to be unity, we have 103·5 as the equivalent of lead, and 8 as that of oxygen. If we follow oxygen further in its combinations, it will be seen that it combines thus:—

| | | |
|---|---|---|
| 8 parts of Oxygen combine with | 1 | part of Hydrogen |
| " " " | 103·5 | " Lead |
| " " " | 20 | " Calcium |
| " " " | 59 | " Tin |
| " " " | 31·7 | " Copper |

The above numbers, therefore, represent the equivalents of the substances to which they are appended.

Again, the equivalent of sulphur is 16, and that weight of sulphur combines with the above weights of hydrogen, lead, calcium, tin, and copper to form sulphides of the respective bases. 35·5 parts of chlorine, or 39·7 parts of selenium, also combine with the same weights, viz. hydrogen 1, lead 103·5, &c., to form chlorides and selenides.

Such compounds are of the simplest class, consisting of single equivalents only; there are, however, many bodies containing more equivalents than two, in which case the following laws are followed.

In one class of compounds the quantity of one of the constituent elements remains constant, while each new compound is formed by the gradual addition of another equivalent of the other constituent element; and it must be borne in mind that no element combines with another in less quantity than its equivalent proportion.

Another series of compounds commences with two equivalents of an element united with some uneven number of equivalents of another element.

In these cases the ratio may be as 2 to 3 and 2 to 5.

The equivalent of a compound body is the sum of the equivalents of the elements forming it; thus, sulphuric acid is composed of one equivalent or 16 parts of sulphur, and three equivalents or 24 parts of oxygen; its equivalent is therefore 40; potash is composed of 39·1 parts of potassium and 8 of oxygen = 47·1. Now, sulphuric acid combines with potash to form sulphate of potash, the equivalent of which is 87·1. In this manner the equivalent of any compound body may be ascertained by adding together the equivalents of the substances forming it.

From what has just been stated concerning the constancy of composition of chemical compounds, we are enabled to calculate the reaction occurring between two or more bodies when decomposition takes place; thus, 87·1 parts of sulphate of potash contain 40 parts of sulphuric acid and 47·1 parts of potash; and if it were desired to obtain sulphate of lead by the decomposition of nitrate of lead by adding to it the above quantity of sulphate of potash, the exact amount of nitrate of lead required could be readily found by adding together the equivalent of the elements forming nitric acid and oxide of lead. Thus, nitric acid is composed of 14 nitrogen and five equivalents of oxygen, or 40, together 54; oxide of lead of 103·5 of lead, and 8 oxygen, together 111·5, which, with the nitric acid, form 165·5. Now, on the addition of 165·5 parts of nitrate of lead in solution to 87·1 parts of sulphate of potash also dissolved in water, 151·5 parts of sulphate of lead will be precipitated in the insoluble form, and 101·1 of nitrate of potash remain in solution.

CHEMICAL SYMBOLS: THEIR USE.—The symbol of an element standing alone signifies one equivalent or atom of the substance; thus, S implies 16 parts of sulphur: a small figure on the right hand side of the symbol indicates the number of equivalents to be represented, as $S_2$, equal to two equivalents, or 32 parts of sulphur.

Two symbols placed thus, FeS, indicate a compound of iron and sulphur, one equivalent of each. Separation of elements by the sign + or a comma, is employed to show the union of two compound bodies, as sulphide of silver and sulphide of lead, which compound may be thus written, $AgS + PbS$, or $AgS, PbS$. A large figure on the same line as the symbol, and on its left side, multiplies the whole of the symbols to the first comma or + sign; thus, $2AgS,PbS$ or $2AgS + PbS$ represents a compound of two equivalents of sulphide of silver, with one of sulphide of lead if, however, it be thus written $2(AgS,PbS)$ it means two equivalents of the whole of the elements which are enclosed in the brackets.

## CHAPTER II.

### PREPARATION OF THE SAMPLE—WEIGHING.

IN all operations connected with assaying, the selection of the sample is the first and most important. It is of little use for the operator to ascertain with the utmost accuracy the percentage of every individual constituent in the mineral operated on, if his sample does not truly represent the average quality of the ore. It should be borne in mind that samples of mineral are generally pieces selected for their richness, or at all events that they represent the most favourable portions of the ore; and no pains should be spared to secure a sample for analysis which will truly represent the bulk of mineral whose value is required to be known.

The assayer should always bear in mind the *object* which his experiments have in view. If they are to ascertain the actual percentage of one or more constituents existing in a certain stone, his labours are comparatively easy,—all that is required being to reduce the *whole* of the specimen to the finest possible state of division, and having well mixed the powder, to analyse a portion of it.

But if it is desired to find out the composition of a mineral or crystal, the greatest possible care must be taken to remove every particle of gangue or other impurities, and to obtain for analysis those portions only of the specimen which represent with greatest accuracy the pure mineral. To effect this, the surrounding rock is first to be removed as carefully as possible, and then the specimen crushed into coarse pieces on a sheet of clean paper. By means of a pocket magnifier and a pair of pincers, clean, typical pieces of the mineral are then to be selected for analysis.

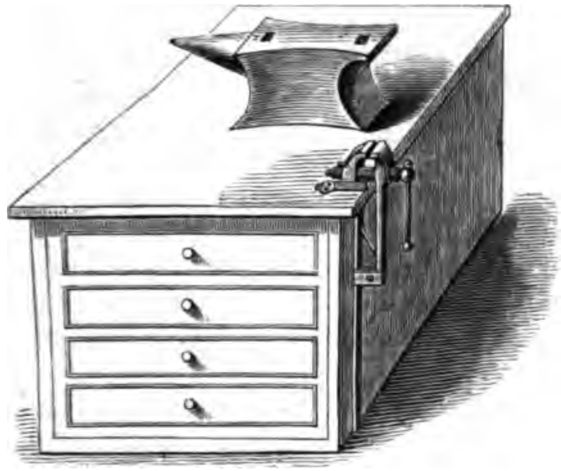
If, however as will most frequently be the case, the object of the assay be to ascertain the average value of a mineral lode or heap of ore, then the assayer must proceed differently. The portion experimented upon must truly represent, in the respective amounts of its valuable material, gangue, quartz, and earthy matters, the great bulk of that of which it professes to be a sample; and this having been secured, the whole must be carefully powdered and passed through fine sieves, taking care that every portion of the mineral goes through. If this be not attended to it will frequently happen that the few grains left out are sufficient to vitiate the whole assay: this is especially liable to be the case when examining ores, the valuable ingredients of which are of a ductile or malleable nature, such as auriferous quartz. In this case it frequently happens that the great bulk of gold exists in the form of one or two small pieces, and these being flattened and beaten out in the operation of powdering will almost certainly be left upon the sieve. In cases like this it is better to collect and assay such pieces separately, and estimate their proportion to the whole weight of the sample, than to attempt to powder and distribute them uniformly.

The ore must always be reduced to a pulverulent form, more or less fine, according to the nature of the chemical operation or assay to which it is to be further subjected. This division is effected by means of the anvil, hammer, pestle and mortar, sieve, method of decantation, or other means generally in use for the preparation of any fine powder. The actual process to be adopted must vary according to the nature of the different bodies under examination. In some cases simple crushing is sufficient; in others the ore will have to be pounded in a mortar; whilst occasionally it is necessary to reduce it to the very highest degree of fineness by elutriation. There is also another operation, which is as strictly mechanical as are the above, viz. washing, dressing, or vanning a sample of ore, the end and aim of which is to separate, in a suitable vessel, by means of water and difference of specific gravity, the earthy or useless, and, in some cases, objectionable portion, from the heavier metallic and valuable portion. This operation is almost always employed on the larger scale in dressing ores for the smelter.

The tools and materials employed are the *anvil* (and stand), *vice*, *hammer*, *files*, *cold chisel*, *shears*, *pestle and mortar*, *steel crushing mortar*, *sieve*, &c.

**The Anvil and Stand** (Fig. 1).—The anvil-stand is constructed of stout wood, about two inches in thickness, and forms a cube of about two feet square. It contains three or four drawers, which serve to hold the hammers, cold chisels, shears, files, &c. which are required in an assay office. In the centre is firmly fixed the anvil, and in one corner a vice may be also firmly secured.

Fig. 1.

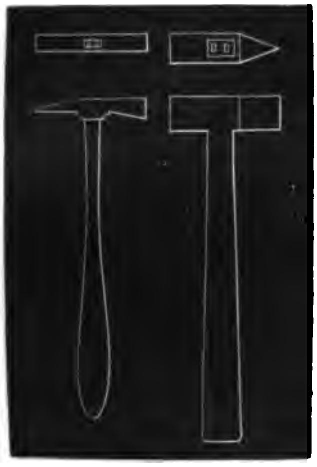


In general the anvil and hammer are employed for the purpose of breaking a small fragment from a mass of ore for examination, or ascertaining whether the button or prill of metal produced in an assay be malleable or otherwise. The anvil is also exceedingly useful as a support for a crucible while breaking it to extract the metallic or other valuable contents.

The anvil is most useful in size when it weighs about 28 lbs.; but one of 14 lbs. will suffice. By reference to the figure, it will be seen that the anvil recommended is of the shape usually employed by the blacksmith.

The hammers, figs. 2 and 3, of which two are requisite, ought to have one end flat and square and the other pick or wedge shaped. The horizontal wedge end of fig. 2 is useful for breaking open crucibles, and detaching small fragments from a specimen of ore the flat end for ascertaining the malleability of buttons of metal. This hammer should weigh about 1 lb. The larger hammer, fig. 3, should weigh about 4 lbs., and is employed for breaking coke sufficiently fine for the use of the furnace, and detaching fragments from refractory minerals, in both of which cases either end may be employed, as may seem most serviceable to the operator. The flat end of this hammer is also used for driving a cold chisel in separating masses of gold, silver, copper, lead, &c. for assay. This hammer has a vertical pick or wedge end.

FIG. 2. FIG. 3.



Very hard and stony materials which have to be broken on the anvil (and all such ought to be so treated) scatter many fragments, to the certain loss of a portion of the substance, and the probable injury of the operator; this can be prevented by wrapping the mineral in a piece of stout brown paper, or, if necessary, in several folds. The fracture can then be safely attempted.

This latter precaution must be specially taken in fracturing gold quartz, or hard rock containing metallic silver, as the loss of a very minute quantity of metal would involve a considerable error in the result afforded by the assay.

All minerals, however, unless very friable, must be reduced to a moderate size—say that of a walnut—by means of the anvil and hammer, before pulverisation; otherwise, if the reduction be attempted in a mortar, it is nearly certain to be injured; moreover, the operator will find his labours much abridged by using the anvil for this purpose.

The anvil can also be made very serviceable in repointing worn or burnt-out tongs, &c. It need scarcely be added that it must be placed as far as possible away from bottles or other frangible articles, otherwise accidents are liable to occur by the forcible projection of fragments of crucibles, stones, &c.

The cold chisel (fig. 4) is employed for cutting off metallic masses for assay. It should be five or six inches long, and about half an inch wide, which is the best size for general use. However, for some purposes, as cutting copper and other very tough metals, it is convenient to have a chisel only a quarter of an inch wide, as these metals are so much more difficult to cut, and the small chisel meets with the least resistance.

FIG. 4.

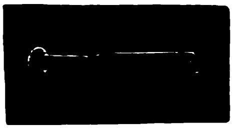
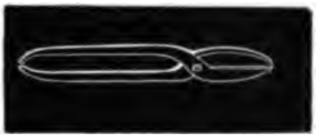


FIG. 5.



Small shears (fig. 5) are also exceedingly useful in cutting off pieces of sheet metal, as lead, for cupellation, scorification, &c.

**THE PESTLE AND MORTAR.**—Mortars are made of various materials, as cast-iron, bronze, porcelain, agate, &c.; the assayer requires one of cast-iron, one of porcelain, and one of agate.

The iron mortar (fig. 6) ought to be of the capacity of from three to four pints; the porcelain (Wedgwood ware)

FIG. 6.

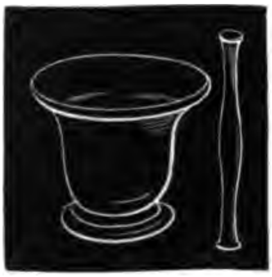
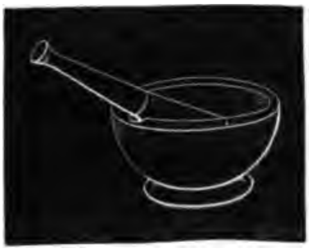


FIG. 7.



(fig. 7) may contain about two pints. The ease with which a mortar may be used, depends much upon its form; and opinion is greatly divided on this subject. Faraday * says that the pestle should be strong, and the size of its superior part such as may be sufficient to allow of its being grasped firmly in the hand, and below to permit a considerable grinding surface to come in contact with the mortar. Its diameter in the lower part may be about one-third or one-fourth of the upper diameter of the mortar. The curve at the bottom should be of shorter radius than the curve of the mortar, that it may not touch the mortar in more than one part, whilst at the same time the interval around may gradually increase, though not too rapidly, towards the upper part of the pestle.

The bottoms of all mortars ought to be of considerable thickness, in order to withstand the smart blows they will occasionally have to receive.

Berzelius recommended (and I have found it extremely serviceable) a mass of pumice-stone for cleansing porcelain mortars. It is used with water as a pestle, and in course of time will be worn to the shape of the mortar; its action will then be more speedy.

Iron mortars can be best cleaned by friction, with a little fine sharp sand, if the ordinary process of washing be not sufficient to remove the adhering substance. Great care must be taken to perfectly dry mortars, especially those of iron, otherwise they will become rusted, and the rust so formed will contaminate the substances pulverised in them.

The iron mortar is principally of use in the reduction of the masses of mineral (broken on the anvil, as before described) to a state of coarse powder, in order to render the substance more readily capable of pulverisation, strictly so called. In the use of the iron mortar, all friction with the pestle ought to be avoided, and the body within it must be struck repeatedly and lightly, in a vertical direction, taking care to strike the large pieces, so that all may be equally reduced. This can be carried on until the whole is about the

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* Chemical Manipulation, p. 149.

size of fine sand; it can then be transferred to the porcelain mortar, where direct blows must be carefully avoided.

The process is now thus carried on: the pestle is to be pressed upon with a moderate force, and a circular motion given to it, taking care every now and then to lessen and enlarge the circles so as to pass over the whole grinding surface of the mortar, and ensure the pulverisation of the mass of mineral submitted to operation. In general, the finer the state of division to which a mineral is reduced, the more accurate and expeditious will be its assay; and in preparing a mineral for assay by the humid method, no labour ought to be spared on this point. Pulverisation is rendered much easier by operating on a small quantity at once, and removing it very often from the sides and bottom of the mortar by means of a spatula. The quantity operated on at one time must be regulated by the hardness or friability of the substance whose pulverisation is to be effected. The harder it is, the less must be taken, and *vice versâ.*

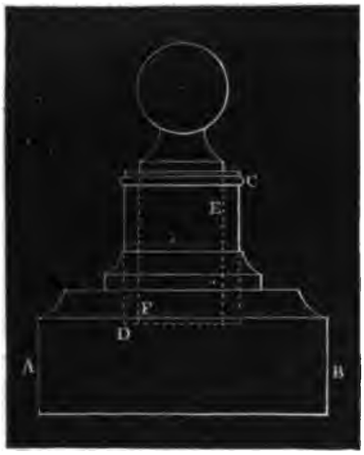
In the use of the iron mortar fragments are occasionally projected. This may be prevented by covering the upper part of the mortar with a cloth. This applies also to the porcelain mortar, for the dust of some minerals has a disagreeable taste and smell. The operator may in some measure protect himself by means of the cloth. Indeed, in some cases, the ambient powder is highly deleterious, as in the pulverisation of arsenical nickel, cobalt, and other ores. Here the simple cloth is not a sufficient protection; it should be slightly damped with water, and tightly tied round the mortar, and firmly held round the pestle, when nothing can escape.

Some minerals can be pulverised with greater ease if they are ignited and suddenly quenched in cold water. Amongst them may be named flint, and many other siliceous matters, as gold quartz. In the pulverisation of charcoal for assays, it will also be found advantageous to ignite it, as hot charcoal is more readily pulverised than cold.

In some instances the powder obtained in the iron or porcelain mortar is not fine enough; recourse should then be had to the agate mortar, in which the mineral, in as fine a state of division as the larger mortars will give it, is ground in small portions at a time, until it is reduced to an impalpable powder.

When small specimens or rare minerals are being operated upon, or if it is especially desirable to avoid loss, it is advisable to use a steel mortar (fig. 8) for the preparatory reduction of the mineral to coarse powder. *A B* and *C D* represent the two component parts of the mortar; these may be readily taken asunder. The substance to be crushed (having, if practicable, first been broken into small pieces) is placed in the cylindrical chamber *e*, *f*; the steel cylinder, which fits somewhat loosely into the chamber, serves as a pestle. The mortar is placed upon a solid support, and perpendicular blows are repeatedly struck upon the pestle with a hammer, until the object in view is attained. (*Fresenius.*)

Fig. 8.



In the selection of agate mortars, they must be examined to see that they have no palpable flaws in them ; very slight cracks, however, that cannot be felt, do not render the mortar useless, although they increase the danger of its destruction by a chance blow.

**The Sieve.**—The operation of sifting is employed when a very fine powder is required, or when a powder of uniform size is needed. Sieves of various materials and different degrees of fineness are necessary. The larger sieve, for preparing coke for the blast furnace, is made of stout iron wire, and must have its meshes from 1 inch to 1½ inch square. The fine coke, which is sifted from that which is the proper size for the blast furnace, may be mixed with that of ordinary size, and employed economically in the muffle furnace. For the preparation of minerals, a set of three sieves should be provided, each one finer than the other. The coarsest may contain 40 holes to the linear inch, the finer or medium sieve 60, and the finest from 80 to 100.

The coarsest sieve is used for preparing galena for assay; the medium, for copper, tin, iron, and other like ores; and the finest, for gold and silver ores, or for preparing any substance for the wet assay, as, in the latter case, the finer the state of division the substance attains, the more rapid will be its solution or decomposition by the liquid agents employed.

The sieve fig. 9 is made of wood, over which is strained in the ordinary manner brass wire-gauze of the necessary degree of fineness. When in use, *B*, fig. 10, is fitted into the lower part of *A* (same figure). This contrivance prevents all loss of the fine powder. If the matter to be sifted be offensive or deleterious to the operator, a sieve termed the drum or box-sieve may be employed (see fig. 10), where *C* represents a cover fitting over the sieve. If small, this may be used in the ordinary way; but if large, its method of use is rather peculiar, and requires some practice to fully develop its powers. One side of the under edge must be held by one or both hands, according to its size, whilst the other rests on a table or a bench. A semi-circular oscillating motion must now be communicated to it by moving the hands up and down at the same time they are being alternately brought into approximation with the sides of the operator.

Fig. 9.

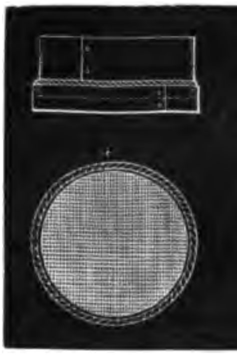
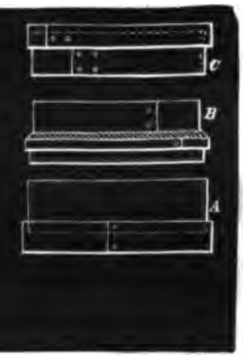


Fig. 10.



In cases of necessity, a sieve may be readily extemporised. Place the powder to be sifted in a piece of fine lawn, or muslin, according to the fineness required, tie it up loosely, and shake or tap the powder, with its muslin or other envelope, on a sheet of paper, and the sifting will be rapidly and easily accomplished.

The sieve is also extremely serviceable in the separation of some ores from their gangues or vein-stones, especially if the latter be stony and hard. This point must be particularly noted, as it is the cause of much variance between the results of different assayers; for instance, part of the same sample of ore might be sent to two assayers, and the produce made by one would be 8½ per cent., and that by the other 9 or 9½, or, in some cases, even more. This discrepancy generally arises from the cause above mentioned. In the one case, the workman has rejected part of the hard gangue, and so rendered the residue richer; whilst in the other he has pulverised the whole, making the produce less, but giving more accurately the amount of metal in the substance submitted to assay.

A knowledge of this fact is also very useful from another point of view; suppose it were wished to separate in a speedy manner, as perfectly as possible, any friable mineral, such as galena or copper pyrites, from its matrix by mechanical means; it might be accomplished by the use of the sieve, as follows: Place a small quantity of the mineral in an iron mortar, and strike, repeatedly, slight vertical blows. When it is tolerably reduced, sift it, and it will be found that what passes through is nearly pure mineral, with only a small quantity of matrix; repeat the pounding and sifting operations, until, after a few repetitions, that which remains in the sieve is nearly pure gangue.

Native metals, as gold, silver, and copper, are also partially separated after the manner above described. The fine particles of metal, during the process of pounding and trituration, become flattened, and refuse to pass through the sieve, whilst the more brittle portions pass through, and are separated.

**Elutriation.**—This process can only be employed for those bodies which are not acted on by water; and it must be remembered that many substances which are usually considered to be insoluble in water, are, when in a very finely divided state, acted upon by this liquid to a more or less extent. The operation is thus effected: The substance is reduced to the finest possible state of division by any of the foregoing processes; it is then mixed with a quantity of water in a glass or other vessel. After a few moments' repose, the supernatant liquid, retaining in suspension the finer particles of the pulverised substance, is poured off, and the grosser parts, which have fallen to the bottom of the vessel, are re-pulverised, and again treated with water. By repeating these processes; a powder of any required degree of fineness may be obtained.

It is seldom, however, that a substance is required for assay by the dry way, in such a minute state of division as is produced by this process. In the humid or wet method it is occasionally very useful. If the supernatant water is roughly decanted off, in certain cases, where the powder to be elutriated is light, the least disturbance of the vessel containing it occasions the distribution of the portion which has settled, throughout the liquid, and the consequent mixture of fine and coarse particles. This can be avoided by the employment of the syphon. The operation is then thus conducted: The syphon is filled with water, and the shorter end placed in the liquid whose transversion is to be effected; the forefinger of the right hand, which, during this time, has been applied to the longer end of the instrument, is now removed, when the water will flow out until it is level with the immersed end of the syphon. Fresh water can then be added, the powder stirred up again, and the operation of decantation by the syphon carried on as long as requisite.

**Washing, Dressing, or Vanning.**—This operation is exceedingly useful for discovering the approximate quantity of pure ore, such as galena, copper pyrites, oxide of tin, native gold or silver, in any sample of earthy matter or ore in which it may be disseminated.

The theory of the operation about to be described is easily understood. Bodies left to the action of gravity in a liquid, in a state of rest, experience a resistance to their descent which is proportionate to their surface, whatever may be their volume and density. Hence it follows, firstly, that of equal volumes the heaviest fall most rapidly; secondly, that of equal densities those having the largest size move with the greatest speed; for in particles of unequal size and like form the weight is proportional to the cube of the dimensions, whilst the surface is only proportional to the square of these dimensions; hence in small particles the surface is greater in relation to the weight than in the large particles; thirdly, that of equal densities and volumes, particles offering the largest surface (those which are scaly or laminated, for example) undergo more resistance in their motion than those which, approaching the spherical form, have less surface. The adhesion of the liquid to the particles of bodies held in suspension is also an obstacle to their subsidence. This force, like the dynamic resistance, is proportional to the surface and independent of the mass or volume, whence it follows that, in a fluid in motion, of bodies having equal volumes, the least dense acquire the greatest rapidity of movement, and are deposited at the greatest distance from the point of departure; whilst with equal densities the smallest grains are carried farthest; and, lastly, with equal densities and volumes the particles exposing most surface traverse the greatest space.

It is, therefore, evident that the most advantageous condition for separating, by washing, two substances of unequal specific gravity or density, is that the heavier shall be in larger grains than the lighter; this unfortunately, however, is a condition that can very seldom be fulfilled, as the heaviest substances are those metallic minerals whose frangibility is nearly always greater than the earthy matters accompanying them as gangues. This being the case, it is very important so to arrange that the fragments of the various mixed substances shall be nearly of the same size. This may be effected by very frequently sifting the mineral during the process of pulverisation, reducing it also more by blows than by grinding, so as to get as little fine powder as possible, as that is nearly certain to be washed away during the process.

The operation of washing or vanning may be performed by one of two methods. In the first, a small stream of running water is employed; in the second, water is added to the substance to be washed, and poured off as necessary.

In the first process, a vessel somewhat resembling a banker's gold scoop (but longer in proportion) is employed; the mineral to be washed is placed in the upper part, and a small quantity of water added, with which the mineral is thoroughly and carefully moistened, and mixed with the fingers. The scoop must then be so inclined that a fine stream of water from any convenient source (say a tap) may fall just above the upper part of the mixture of mineral and water; then, firmly holding the larger and upper end of the scoop with the left hand, and sustaining the lower part with the right, it is shaken frequently in the direction of its longitudinal axis. At each shake, all the particles in the scoop are so agitated that they become suspended in the water, and the current of liquid running from the tap into the scoop moves them all in its own direction; but they are deposited at different distances from the point at which the water enters, the heaviest being carried through but a very small space. It is now soon seen that the mineral assumes a heterogeneous surface; at the upper part, the heavy portions are seen nearly pure; the light substances, on the other hand, are nearly without mixture at the lower end, and in the intermediate part the heaviest portion of the mixture is nearest the upper end. If the washed matter were now to be divided into horizontal layers, the heaviest matter would be found at the bottom, and the lightest on the surface. Things being in this state, the scoop must be made to oscillate on its axis, so that the latter remain immovable, and in a slightly inclined position. In this manner, the layer of water running over the surface of the mineral agitates that part only, and carries off all light substances there deposited in the previous operation. When necessary, these matters may be removed by the finger, and made to run into a vessel placed below the scoop, in which all the water and matters carried off are received. This operation, however, must not be hurriedly performed, so as to mix the parts already separated: each layer must be removed separately, commencing with the upper one. This being done, the scoop must be alternately kept in motion by shakings, as at first, and then on its axis, and the washing off of the finer particles renewed, and so on until the separation is effected as far as may be judged necessary.

At the commencement of the operation, the water carries out of the scoop the lightest particles, as organic matter, clay, &c.; at a little later period the water carries with it a small but definite quantity of the heavier portion, the proportion of which increases as the operation proceeds, until at last the greatest possible care is required. It is always better to rewash the latter portion which passes off from the scoop; hence the necessity of allowing all the wash-water passing from it to collect in a vessel placed for that purpose.

In the second method of washing, a tin, zinc, or wooden pan is employed. It should be circular, one or two feet in diameter and three or four inches deep; the sides should descend in a conical manner, so that the bottom is not more than four inches in diameter, and the angle between it and the sides as sharp as possible.

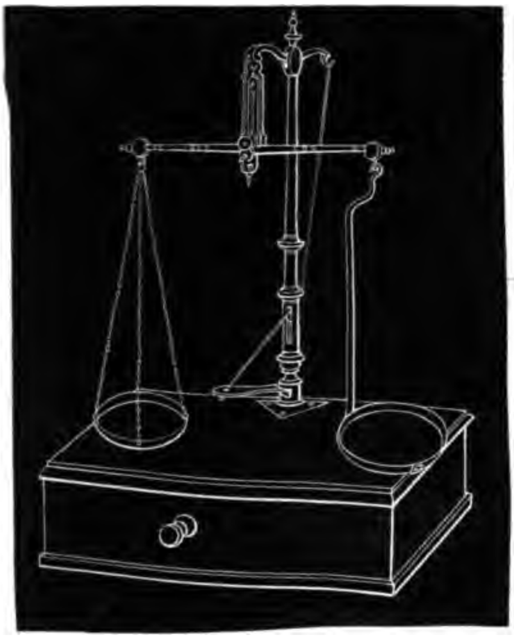
The substance to be examined is placed in the washing-dish, the latter filled with water, and the mineral well mixed with it until perfectly moistened, as before. After a moment or so the muddy water is poured off, and the operation repeated until the water passes off clear. When this happens, only so much water must be placed in the pan as will leave a slight layer on the mineral. Now, by holding the pan in one hand, and shaking it with the other, the greater part of the heavy mineral, gold or otherwise, will fall below the sand. If now the pan be inclined towards the hand which is shaking it, the lighter portions, even if tolerably large, will flow off with the water, leaving the heavier matters in the angle, from which, with ordinary care and a little practice, it is difficult to disturb them. If there be a large quantity of earthy matter, this may be (after sufficient shaking) removed by the finger, as in the first described process. By careful repetitions of these processes, the whole, or nearly the whole, of the sandy and earthy matters may be removed, and the gold or other mineral left nearly pure. This is the plan employed in *prospecting* for gold, diamonds, and other gems, and, in some cases, for their commercial extraction.

In Cornwall, and other mining counties, this operation is very cleverly and carefully performed on the miner's common shovel, and the richness of any particular sample of either tin, lead, or copper is thereby determined with a very near approach to accuracy.

## THE BALANCE.

**Operation of Weighing.**—At least three balances will be required in a laboratory where general assays are performed. The first must be capable of carrying three or four pounds in each pan, and must turn with a quarter of a grain. This may be of the form of the bankers' or bullion balance (fig. 11), and may be employed in weighing samples of gold quartz or silver ore containing metallic grains capable of being separated by the sieve (see p. 18); the second (fig. 12), or rough assay balance, is similar to the apothecary's scales; it should take 1000 grains in each pan, and turn with one-tenth of a grain. This serves for weighing samples of ore and fluxes for assay, and for determining the weight of buttons or prills of lead, tin, iron, copper, &c. obtained in an assay.

Fig. 11.



The third and most delicate, or true *assay balance* (fig. 13) should carry about 1000 grains; must turn distinctly and accurately with the $\frac{1}{1000}$th of a grain. This is employed in the assay of gold and silver bullion, and in the assay

Fig. 12.

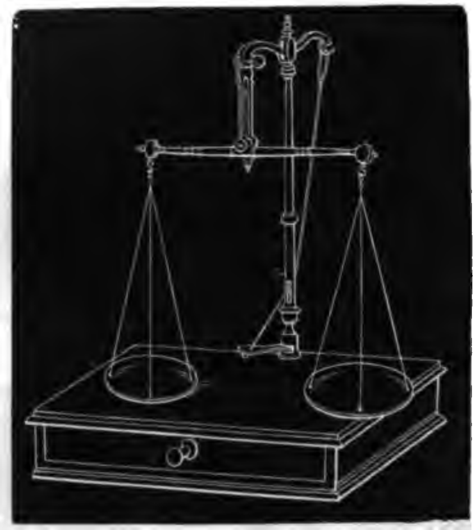
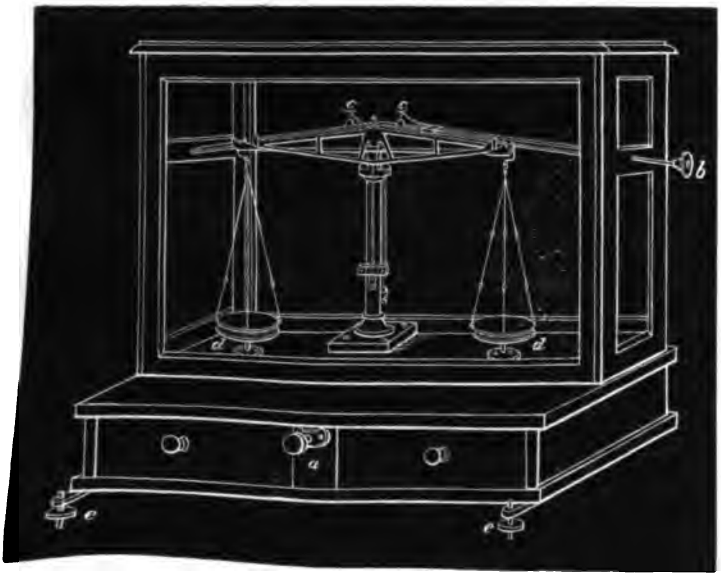


Fig. 13.



of minerals containing gold and silver; also for general analytical purposes. The first two balances may be used, with ordinary care, by anyone; but the third balance, in its use and adjustment so as to maintain its extreme accuracy, requires some particular instructions, which necessarily involve the principle of the balance. These have been so admirably given by Mr. Faraday, in his 'Chemical Manipulations,' that we can do no better than transcribe them.

'The theory of this balance is so simple that the tests of its accuracy will be easily understood and as easily practised. It may be considered as a uniform inflexible lever, supported horizontally at the centre of gravity, and supporting weights at equal distances from the centre by points in the same horizontal line with the centre of gravity. If the weights be equal the one will counterpoise the other; if not the heavier will preponderate. In the balance, as usually constructed, there are certain departures from the theory as above expressed—some from the impossibility of execution, and others in consequence of their practical utility; and a good balance may be said to consist *essentially* of a beam made as light as is consistent with that inflexibility which it ought to possess, divided into two arms of equal weight and length by a line of support or axis, and also terminated at the end of each arm by a line of support, or axis, intended to sustain the pans. These three lines of support should be exactly parallel to each other in the same horizontal plane, and correctly perpendicular to the length of the beam; and the plane in which the line should be raised more or less above the centre of gravity of the beam, so that the latter should be exactly under the middle line of suspension. It will be unnecessary in this place to speak of the coarse faults which occur in the ordinary scales—these will be easily understood; and from what has to be stated of the examination of the most delicate instrument, the impossibility of avoiding them without incurring an expense inconsistent with their ordinary use will be as readily comprehended.'

Two principal things have to be attended to in the selection of a balance—its *accuracy* and its *delicacy*. The accuracy depends upon the following conditions.

1. *The arms should be equal to each other in length.* The length of each is accurately the distance from the middle to the distant knife edge, all the edges being considered parallel to each other, and in the same plane. The two arms should accord perfectly in this respect. This equality may be ascertained in two or three ways. Suppose the balance with its pans to vibrate freely, and rest in a horizontal position, and that after changing the pans from one end to the other, the balance again takes its horizontal state of rest; in such a case, an almost certain proof is obtained of equality in length of the arms. They may, however, be equal, and yet this change of the pans from end to end may occasion a disturbance of equilibrium, because of the unequal distribution of weight on the beam and pans; but to insure an accurate test, restore the pans, and consequently the equilibrium, to the first state: put equal, or at least counterpoising weights into the pans, loading the balance moderately, and then change the weights from one pan to the other, and again observe whether the equilibrium is maintained; if so, the length of the arms is equal.

Equality of weight is not so necessary a condition, although this should be obtained as accurately as possible. One arm with its pan may be considerably heavier than the other, but from the disposition of the weight in the lighter arm towards the extremity, or in the heavier towards the middle of the beam, the equilibrium may be perfect, and therefore no inaccuracy be caused thereby in the use of the balance. Instruments are usually sent home in equilibrium, and require no further examination as to this particular point than to ascertain that they really are in adjustment, and that after vibrating freely they take a horizontal position.

2. *The beam must be of such a form and strength that it will not bend when loaded with the greatest weight the balance is intended to carry.* All well-made modern balances are sufficiently rigid in this respect, and may be safely trusted to carry their full weight without flexure of the beam. It should also be as light as practicable.

3. *The knife edges supporting the pans, and the centre one on which the beam vibrates, must be accurately in the same line.*

The *delicacy* of a balance likewise depends upon several conditions.

The centre of gravity *must be very little below the fulcrum.* If it be considerably depressed, then, upon trying the oscillations of the balance by giving it a little motion, they will be found to be quick, and the beam will soon take its ultimate state of rest; and if weights be added to one side, so as to make it vibrate, or to bring it to a certain permanent state of inclination, the quantity required will be found to be comparatively considerable. As the centre of gravity is raised the oscillations are slower, but producible by a much smaller impulse; the beam is a longer time before it attains a state of rest, and it turns with a smaller quantity.

If, however, the centre of gravity coincides with the fulcrum or centre of oscillation, then the balance is said to *set*, that is, the smallest possible weight will turn the beam; the oscillations no longer exist, but one side or the other preponderates with the slightest force, and the valuable indication which is furnished by the extent and velocity of the vibrations is lost.

The case in which the centre of gravity is above the fulcrum rarely if ever occurs. Such a balance, when equally weighted, would *set* on the one side or the other, that side which was in the slightest degree lower tending to descend still further, until obstructed by interposing obstacles.

In balances intended to carry large quantities (as in the balance for weighing gold quartz, &c.) it is necessary to place the centre of gravity lower than in those for minute quantities, that they may vibrate regularly and readily. This is one cause why they are inferior in delicacy, for, as a consequence of the arrangement, they will not turn except with a larger weight.

Balances are also liable to *set* when overloaded. Thus, if a balance be equally weighted in each pan, but overloaded, it will, if placed exactly horizontal, remain so, but the slightest impulse or depression on one side destroys the equilibrium; the lower side continues to descend with an accelerated force, and ultimately remains down, being to all appearance heavier than the other. Generally speaking, the more delicate a balance the sooner this effect takes place; this is one limit to the weight it can properly carry.

The vibrations of a balance vary with the quantity of matter with which it is loaded: the more the weight in the pans, the slower the vibration. These should be observed, and the appearances retained in the mind, in consequence of the useful indications they afford in weighing. A certain amplitude and velocity of vibration would indicate to a person used to the instrument, nearly the weight required to produce equilibrium; but the same extent and velocity, with a weight much larger or smaller, would not be occasioned by an equal deficiency or redundancy of weight, as in the former case.

The weight also required to effect a certain inclination of the beam, or to turn it, should be known, both when it is slightly and when it is heavily loaded. Thus, if the instrument turns with $\frac{1}{1000}$th of a grain, with 1000 grains in each pan, or with $\frac{1}{1000000}$th of the weight it carries, it may be considered perfect.

The friction of the knife edges must be as slight as possible.

Most of the faults in the working of a balance, if ordinarily well made, depend upon imperfections in the middle knife edge and the planes upon which it rests.

The edge is made either of agate or steel, preferably the former, and should be formed out of one piece, and finished at once, every part of the edge being ground on the same flat surface at the same time. In this way the existence of the two extreme or bearing parts of the edge in one line is insured; but when the two parts which bear upon the planes are formed separately on the different ends of a piece of agate or steel, or, what is worse, when they are formed on separate pieces, and then fixed one on each side the beam, it is scarcely possible they should be in the same line; and if not, the beam cannot be correct. These knife edges usually rest on planes, or else in curves. The planes should be perfectly flat and horizontal, and exactly at the same height; the curves should be of equal height, and their axes in the same line. If they are so, and the knife edge is perfect, then the suspension will be accurately on the line of the edge, and reversing the beam will produce no change. The balance must always be kept perfectly level by means of the three screws on which it stands, and adjusted by the spirit-level or plumb-line with which it is furnished.

The balance should be kept in a well-lighted dry room, quite away from acid or other vapours. The case should be kept closed as much as possible, and a glass vessel full of lumps of good quick-lime should be kept in it. When the lime falls to powder it should be renewed.

In order to test the accuracy and delicacy of a balance, remove the pans and their end supports, and notice how the beam oscillates. When it has been found to oscillate with regularity, and gradually to attain a horizontal position of rest, it should be reversed—that is, taken up and turned half-way round, so as to make that which before pointed to the right now point to the left. The beam should then again be made to oscillate, and if it perform regularly as before, finally resting in a horizontal position, it has stood a severe test, and promises well. Then replace the pans and repeat the tests, noticing the time required for each oscillation. When the pans are hung upon the beam, the balance should of course remain horizontal. They should be tried by changing then by reversing the beam, and afterwards by changing the pans again. The pans are best suspended by very thin platinum wire, so as to avoid hygrometrical influence upon them.

Afterwards load the balance with the full weight it is intended to carry—say 1000 grains in each pan, and notice if the indications are as rapid upon adding or subtracting the smallest weight as they were when the pans were empty.

Tests of this kind are quite sufficient for the purpose of the assayer, who, having ascertained that his balance, whether slightly or fully laden, vibrates freely, turns delicately, has not its indications altered by reversing the beam or changing counterpoising weights, may be perfectly satisfied with it.

The irregularities which may be discovered by these tests are best corrected by a workman; but as in all the best balances now made adjusting screws for these purposes are provided, it has been thought advisable to introduce here such matter as, after careful perusal, will enable every one to adjust and examine his balance properly; so that, in the absence of a skilled workman, it may without much danger be put into working order by the assayer himself, if accidentally damaged by rough treatment.

**The Weights.**—Various kinds of weights are necessary for the different balances required by the assayer. For the larger balance, Troy-weights from 4 lbs. to $\frac{1}{4}$ grain will be requisite; for the second size, weights from 1000 grains to $\frac{1}{10}$th part of a grain; and for the assay balance, weights from 1000 grains to $\frac{1}{1000}$th of a grain.

The best material adapted for weights is unquestionably platinum. This is, however, too expensive for its general adoption, and therefore brass weights are almost invariably employed down to the ten or twenty grain weight, the smaller ones only being of platinum. On the Continent weights are generally made of silver, and if of brass are electro-gilt. For the smallest weights of all, those below 0·10 grain, aluminium is often used, its lightness, and consequently greater bulk, enabling these small weights to be made considerably larger than if they were of platinum. The riders are generally of silver-gilt wire. The slight tarnish which gradually forms on brass weights may be disregarded until it becomes very thick. Weights ought never to be touched with the fingers, and should, when not in use, be kept tightly fastened in their box, away from all acid fumes. The most convenient series in which to have the weights is 600, 300, 200, 100, 60, 30, 20, 10, 6, 3, 2, 1, ·6, ·3, ·2, ·1, &c. This is preferable to the series formerly employed, as it admits of the use of a less number of weights to arrive at any required amount.

Peculiar weights are necessary for the assay of gold and silver bullion in England (with the exception of assays for the Bank of England; see Gold assay), gold being reported in carats, grains and eights, and silver in ozs. and dwts. The most convenient quantity of either of the precious metals for assay is 12 grains. The quantity taken, however, is of no very great consequence; but whatever its real weight, it is denominated in England the *assay 'pound.'* This assay 'pound' is then subdivided into aliquot parts, but differing according to the metal. The silver assay 'pound' is subdivided, as the real Troy pound, into 12 ounces, each ounce into 20 pennyweights, and these again into halves (the lowest report for silver), so that there are 480 different reports for silver, and therefore each nominal half-pennyweight weighs $\frac{1}{40}$th part of a Troy grain, when the 'pound' is 12 grains.

*Assay Weights for Silver.*

| Silver ozs. | dwts. | grs. | Assay grains |
|---|---|---|---|
| 12 | 0 | 0 | 12 |
| 11 | 0 | 0 | 11 |
| 6 | 0 | 0 | 6 |
| 3 | 0 | 0 | 3 |
| 2 | 0 | 0 | 2 |
| 1 | 0 | 0 | 1 |
| 0 | 10 | 0 | 0·500 |
| 0 | 5 | 0 | 0 250 |
| 0 | 3 | 0 | 0·150 |
| 0 | 2 | 0 | 0·100 |
| 0 | 1 | 0 | 0·050 |
| 0 | 0 | 12 | 0·025 |

The *gold assay 'pound'* is subdivided into 24 carats, each carat into 4 assay grains, and each grain into eighths, so that there are 768 reports for gold, and the assay 'pound' weighing 12 Troy grains, the lowest report, or $\frac{1}{8}$th assay grain, equals $\frac{1}{64}$th Troy grain; thus

*Assay Weights for Gold.*

| Gold carats | grs. | eighths | Assay grains |
|---|---|---|---|
| 24 | 0 | 0 | 12 |
| 22 | 0 | 0 | 11 |
| 12 | 0 | 0 | 6 |
| 6 | 0 | 0 | 3 |
| 3 | 0 | 0 | $1\frac{32}{64}$ths |
| 2 | 0 | 0 | 1 |
| 1 | 0 | 0 | $\frac{32}{64}$ths |
| 0 | 2 | 0 | $\frac{16}{64}$ths |
| 0 | 1 | 0 | $\frac{8}{64}$ths |
| 0 | 0 | 6 | $\frac{6}{64}$ths |
| 0 | 0 | 3 | $\frac{3}{64}$ths |
| 0 | 0 | 2 | $\frac{2}{64}$ths |
| 0 | 0 | 1 | $\frac{1}{64}$th |

In cases where the very smallest weights have to be employed, great care must be taken in seizing them with the forceps, as they are apt to spring away and be lost. In the assay balance (fig. 13), the use of weights less than $\frac{1}{10}$th of a grain is avoided by a very ingenious contrivance. Each side of the beam is equally divided into ten parts, and over the beam on either side is placed a sliding rod, as represented in the figure. The object of these rods is to carry, in the direction of the beam, the small bent piece of wire, (letter *c*, fig. 13,) called a *rider*, which serves in lieu of the smallest weights—the $\frac{1}{100}$th and the $\frac{1}{1000}$th. These riders are thus employed: one weighing $\frac{1}{10}$th of a grain is placed on the cross-piece of the extremity of the sliding rod just mentioned, and the rod thus furnished is brought gradually along the beam from the centre to the end, until the rider can be deposited on the division on the beam marked 10; the balance is then loaded on that side with a weight equal to $\frac{1}{10}$th of a grain. If now the rod be advanced to the centre of the balance, and the rider dropped on the mark 5, the half of $\frac{1}{10}$th of a grain will be pressing on that side of the balance, or, in other words, ·05th of a grain; and when the rider is at the marks 1, 2, 3, 4, respectively, ·01, ·02, ·03, ·04 of a grain will be indicated. With a rider weighing $\frac{1}{100}$th of a grain, thousandths of grains may be indicated; thus the last rider placed on the marks 1, 2, 3, 4, would equal ·001, ·002, ·003, ·004 grain, &c.

**The Method of Weighing.**—The operation of weighing is very simple; but as in the hands of the chemist and assayer it becomes one of great frequency, the facilities for its performance require to be mentioned. It should in the first place be ascertained before every operation that the balance is in order, so far as relates to its freedom of vibration, and also that no currents of air are passing through the case, so as to affect its state of motion or rest, a situation being chosen where such influence may be avoided. In most cases there is a small projecting arm on the upper part of the beam, which, being turned either to the right or left hand side of the beam, as required, serves to establish perfect equilibrium. Perfect equilibrium is, however, a matter of no consequence if the assayer observes one or two simple rules. He should never on any account *weigh* by the *direct* method; that is, he should never obtain the weight of a substance by putting it at once into one pan, and then counterpoising it by adding weights to the other pan. This method is only to be relied on when the balance is of rare perfection, and is used by no one but the assayer himself. The plan of weighing by difference should invariably be adopted. By this means the weight of any body can be readily ascertained, no matter whether the arms of the balance are of equal length or the pans are in equilibrium.

In the first place, it should be a rule that one pan, preferably the left, be reserved for the substance to be weighed, and the other pan be set apart for the weights.

Supposing the weight of a portion of mineral is required. First place a clean watch glass, or platinum capsule, in the left pan, and carefully ascertain its weight. Let us suppose it weighs 106·347 grains; now put the mineral in the watch-glass and ascertain the united weight of the two. This we will imagine comes to 763·776. By subtracting the weight of the glass or capsule from this we find the true weight of the mineral, which is 763·776 − 106·347 = 657·429. The substance to be weighed must *never* be put direct into the pan. By weighing in this manner by difference the errors arising from inequality in the equilibrium or length of arms are eliminated.

Nothing should ever be weighed until it is perfectly cold. It is also inadvisable to weigh anything immediately after it is taken from a cold place to a warmer one, as the substance in such case will act as a hygroscopic body, and by condensing moisture, will appear heavier than it really is.

Powders are conveniently weighed by filling a small stoppered tube bottle with them, then weighing the whole, and after pouring out the requisite amount of its contents, re-weighing the bottle and powder. The difference gives the weight of powder used. This is a very convenient plan if several portions of the same substance are required for different analyses. The tube will require re-weighing each time after the quantities required for each analysis are shaken out into the receptacles.

A delicate balance is always furnished with means of supporting the pans independent of the beam; and the beam itself is also supported when required by other bearings than its knife edges, and in such a manner as to admit of the rapid removal of these extra supports, that the instrument may be left free for vibration. This is done that the delicate edges of suspension may not be injured by being constantly subjected to the weight of the beam and the pans, and that they may suffer no sudden injury from undue violence or force impressed upon any part of the balance. When, therefore, a large weight of any kind is put into or removed from the pans, it should never be done without previously supporting them by these contrivances; for the weight, if dropped in, descends with a force highly injurious to the supporting edges; also if a large weight be taken out without first bringing the pans to rest, it cannot be done without producing a similarly bad effect.

The weights should not be put into the pan at random. It is a mistake to suppose that time is saved by such a plan. The highest probable weight should be added first, and then the set should be gone through systematically down to the smallest weight, retaining or removing each weight in order according as it is too little or too much. The exact weight of a body will be found in this manner in far less time than would be required were the weights added by guess.

When a weight is put in which is assumed to be nearly equal to the substance to be weighed, the balance should be brought to a state of rest, and should then be liberated gradually by turning the handle, so as to leave the pans wholly supported by the beam. The whole being on its true centres of suspension, it will be observed whether the weight is sufficient or not; and the rapidity of ascent or descent of the pan containing it, will enable a judgment to be formed of the quantity still to be added or removed.

Great care should be observed in recording the weight in the note-book. The weight should first be ascertained from an inspection of the vacancies in the box of weights, and then verified by an examination of the weights themselves. This is conveniently done whilst replacing them in the box, which should be done immediately after each weighing.

In some cases, where great accuracy is not of so much importance as rapidity in getting out approximate results, a plan may be adopted recommended by Mr. F. F. Mayer in the American 'Journal of Science and Art' for 1861.

Mr. Ch. Mène, of Creusot, gave (in the Journ. de Pharm. et de Chimie, for October, 1858) a mode of weighing which does away to a great extent with the tediousness and difficulties attending the drying of many precipitates. He washes the precipitate thoroughly by decantation, and then introduces it carefully into a bottle the exact weight of which when filled with distilled water at a certain temperature is known. Since the precipitate is heavier than water, the bottle when filled again will weigh more than without the precipitate, and the difference between the two weights furnishes the means of calculating the weight of the precipitate.

In case the precipitate settles but slowly, it may be collected on a filter, and, together with a filter, after washing, be introduced into the bottle, in which case the weight of the filter and its specific gravity, supposing any difference should exist between its own and that of water, is to be taken in account. Precipitates soluble in or affected by water may be weighed in some other liquid.

Mr. Mayer has applied this principle on a large scale as far back as 1855.

In that year he was engaged in the manufacture of carbonate of lead from refuse sulphate of lead, by treating the latter, in a pulpy condition, with carbonate of soda. The sulphate of lead used contained very varying proportions of water and soluble impurities; from which latter it had first to be freed by washing. It was then in the state of a thin pulp, and the difficulty was to find the amount of the dry sulphate of lead, as it was a matter of importance to use a little carbonate of soda, and to obtain as pure a carbonate of lead and sulphate of soda as possible. This could only be done by weighing it as whole or in portions; but as the drying of a tubful of sulphate of lead (from 500 to 1200 lbs.) was impracticable, and sampling not less so, since the upper strata contained a much larger proportion of water than the lead at the bottom, the following method was contrived, which enabled the management of the process to be left in the hands of a workman :—

A strong oaken pail was taken, weighing 8 lbs. when empty, and a black mark was burnt in horizontally around the inside of the pail two inches below the rim, up to which mark it held 20 lbs. of water. The specific gravity of sulphate of lead being 6·3, the pail, if filled up to the mark, would hold 126 lbs. of pure sulphate of lead. The specific gravity of water being 5·3 less than that of sulphate of lead, it followed that if there were 1 lb. of water in the pailful of moist sulphate, the pail would weigh 5·3 lbs. less than 126 (+8, the tare of the pail)=120·7(+8); if there were 2 lbs. of water present, the weight would be 115·4(+8), and so on. This enabled a table to be calculated giving in one column the actual weight of the pail when filled with moist sulphate, and opposite, in a second column, the amount of dry sulphate corresponding to the gross weight. The weight of dry sulphate was thus found as accurately as could be desired, although the amounts varied in practice from 30 to 105 lbs.

This is nothing but an application of the Archimedean theorem, that when a solid body is immersed in a liquid, it loses a portion of its weight, equal to the weight of the fluid which it displaces or to the weight of its own bulk of the liquid.

This is precisely the principle applied by Mr. Mène. The precipitate he obtains by a certain chemical manipulation, is a substance of known composition and specific gravity. Supposing it to be sulphate of lead, and the bottle when filled with water at the normal temperature to weigh 70 grammes = 50 grammes of water and 20 for tare. After introducing the precipitate and filling again with water it weighs 71·06 grammes. Now as the specific gravity of sulphate of lead is 6·3, or as the weight of a cubic measure of sulphate of lead is 6·3 times that of a cubic measure of water, and as the space of one part by weight of water is taken up by 6·2 parts by weight of sulphate of lead, it follows that the quantity of the sulphate of lead in the bottle, which has taken up the space of one part by weight of water, increases the original weight of the bottle (filled with pure water) by 5·3. To find the amount of water displaced it is only necessary to divide the overweight (1 06 grammes) by 5·3=0·2, which added to the overweight 1·06 + 0·2 gives 1·26 grammes as the weight of the precipitate.

Hence the rule, which is of great convenience in volumetric analysis, that to find the weight of a moist precipitate which is a compound of known specific gravity, weigh it in a specific gravity bottle or some other vessel of known weight when filled with water, or any other liquid at the normal temperature; again fill it with the water or other liquid, divide the excess of the new weight by the specific gravity of the substance, less that of the water or other liquid (that of water being=1), and add the quotient to the overweight, which gives the weight of the precipitate.

The principle exemplified by Mr. Mayer may not be novel; but as it has never been fully exemplified before, chemists and assayers, as well as manufacturers, especially of colours, will probably also find it of interest, and certainly highly practicable and easy of execution.

# CHAPTER III.

## GENERAL PREPARATORY CHEMICAL OPERATIONS.

**Calcination.**—Strictly speaking the term calcination means the production of an oxide or *calx* by combustion, and it necessarily involves the intervention of atmospheric oxygen. But in a metallurgical sense the term is restricted to the separation of any volatile matter from a mineral substance by the aid of heat alone, the atmosphere being totally or partially excluded, or the production of rapid changes of temperature, so as, for instance, to render minerals more fragile by quenching in water, &c.

Thus, we speak of the calcination of minerals, as iron, or zinc, ores, &c., whose matrices are argillaceous, to expel water; and also of gypsum to expel water; the carbonates of lime, iron, copper and lead, are calcined to separate carbonic acid; the hydro-carbonates of zinc and iron, to get rid of both water and carbonic acid; cobalt, nickel ores, &c. to separate arsenic and sulphur; the iron ore found in the vicinity of collieries, to expel bituminous matter; and wood and bones to expel volatile organic matter. Where the operation is accompanied by combustion, and requires the oxygen of the atmosphere, it is termed roasting.

Crucibles are conveniently used in calcination, as no stirring of the mass is required. They may be made of various materials, as clay, plumbago, platinum, silver and iron. Silver cannot be employed at a heat greater than dull redness. The selection of the crucible must depend upon the substance under operation; they must all be furnished with covers.

In almost all operations in assaying it is necessary to estimate the amount of volatile matter lost by calcination.

A very high temperature is seldom required in calcination;

usually an air furnace will give enough operation is finished, the crucible must be rem fire and allowed to cool gradually. When com remove the cover and take out the contents by m spatula. If any adhere, a small brush will be fou useful for its removal. The difference in weight before after calcination will represent the volatile matter.

When the substance to be calcined is fusible, the crucible and contents must be weighed before ignition; and the loss of weight is equal to the quantity of volatile matter expelled; in fact, this latter is usually the most satisfactory method of conducting the experiment.

If the ignited or calcined substance be soluble in water, it can be removed from the crucible by that menstruum, employing heat if required; if not, any suitable acid may be used.

If the substance to be calcined decrepitates on heating, it must be previously pulverised and heated slowly and gradually in a well-covered crucible.

Certain substances, as carbonate of lead, undergo a material alteration by contact with the gases given off during the combustion of the fuel in the heating furnace; others, such as carbonaceous matters, are consumed by the introduction of atmospheric air. All such substances must be calcined in a closely-covered crucible placed in a second crucible (also covered) for further protection.

In some rare cases, however, these precautions are not sufficient. In such, either a weighed porcelain or German glass retort must be employed.

Sometimes earthenware crucibles lined with charcoal are employed in calcination; for even if the substance be fusible it may generally be collected and weighed without loss, as very few bodies either penetrate into or adhere to a charcoal lining. In this way grey cobalt and other arsenio-sulphides are calcined at a high temperature to expel the greatest possible amount of arsenic and sulphur.

The selection and proper management of crucibles will be given in the next chapter.

**Roasting.**—In this operation carbon, sulphur, selenium, antimony and arsenic are separated from certain metals with which they were combined. Roasting differs from calcination in this particular, the latter is carried on in close vessels, independent of the atmosphere, the former in open vessels by the aid of the atmosphere. It is thus we are enabled to separate the bodies just mentioned by this process; for the oxygen of the air, by combining with them, forms a volatile substance which the heat expels. Thus, in roasting sulphide of copper and iron (copper pyrites), the sulphur, copper, and iron mutually combine with oxygen to form sulphurous acid (volatile), protoxide of copper and peroxide of iron, thus:—

$$2(FeS + CuS) + 13O = Fe_2O_3 + 2(CuO) + 4(SO_2).$$

This is the final change in this case. During the process, however, some sulphate and sub-sulphate of copper and iron are formed. This change will be given under the head of Copper Assay.

When carbonaceous matters are roasted, the operation also takes the name *combustion*, or incineration; because the object of roasting a fuel, for instance, is generally to ascertain the amount of ash left.

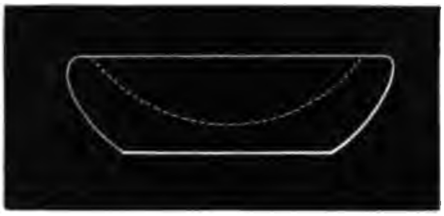
In roasting, in the ordinary acceptation of the term, the body must *not be fused*, but kept in a pulverulent state; but there are some cases in which fusion is allowable, as in *cupellation* and *scorification*.

The process of roasting is performed in different ways; in one, a small flat vessel, called a roasting test (fig. 14) is employed, made of the same material as the earthen crucibles, and similar to a saucer. It is most conveniently heated in a muffle. The substance to be roasted must be finely pulverised, placed in the roasting vessel, and constantly stirred with an iron or glass rod until no fumes are given off, or until it ceases to evolve the odour of sulphurous acid, when sulphur is one of the constituents to be eliminated.

The operation may also be performed in a crucible, in which case it must be inclined to the operator, so that the draught of air passing to the furnace flue may impinge as much as possible on the substance under manipulation.

During roasting the heat must be carefully regulated for some time. At first it ought only to be the dullest red; and the substance must be assiduously stirred in order to present the largest possible surface to the action of the atmosphere, and prevent fusion, for some assays, when roasting, will fuse readily at a low temperature unless the surface be continually renewed. Even by paying the utmost attention to this point it cannot be always prevented, as for instance when sulphide of antimony is being roasted. In these cases the assay must be mixed with its own weight of powdered quartz or fine white sand (silver sand): the operation will then proceed steadily.

FIG. 14.



If the assay at all agglutinates it must be taken from the fire, and rejected if the substance be plentiful; if not, the fused mass must be carefully removed from the crucible or test, pulverised, and the roasting recommenced. In this case, however, the operation is always very tedious, and the final result less exact, so that great care ought to be taken at the commencement of the roasting.

When the assay has been kept at a dull red heat for some time, and shows no signs of agglutination, the heat may be slightly increased; at the same time stirring must be diligently pursued. After the heat has arrived at full redness there is little fear of fusion; and as the operation proceeds more rapidly at a high temperature than a low one, it is well now to increase the heat to a yellowish red, and even in certain cases to nearly a white heat. If the stirring of the assay has been constant during the various gradations of heat, the roasting at this point will be accomplished; and the remaining operations of the assay may be proceeded with.

This is the general plan of operation: but different substances require for roasting a different degree of heat; for instance, copper pyrites requires a higher temperature than grey copper ore, and the heat employed must in every case be adapted to the substance to be roasted. Some substances, for instance, arseniates, sulphate of lead &c., cannot be roasted by heat alone. These require the addition of a carbonaceous body to remove the combined oxygen and allow the arsenic, sulphur, &c. to be completely roasted off. Carbonate of ammonia, in some cases, is also added to the mixture to separate the sulphates formed during the roasting of sulphides.

In cases where the metallic bases of the sulphides are volatile, either as such, or as oxides, as, for instance, galena, sulphide of antimony, &c., a loss of metal will always result during the roasting process.

It may be as well to mention here, that platinum capsules are useful in certain roasting operations. The sulphide of copper, iron, and molybdenum, are conveniently oxidised in this kind of vessel, without much fear of injury, provided fusion of the roasting substance be carefully avoided. Platinum vessels must also be used in ascertaining the amount of ash in coal where the experiments are required to afford exact results.

**Reduction.**—The process of reduction consists in removing oxygen or analogous element from any body containing it, usually by means of either carbonaceous matter or hydrogen, or a body containing both of these elements, and leaving the metal behind, usually in the form of a melted button. The *rationale* of the operation is as follows, when oxide of lead is reduced with carbon:—

$$2(PbO) + C = 2Pb + CO_2.$$

In this first case we start with oxide of lead and carbon, and as a result we obtain metallic lead and carbonic acid.

The reaction between oxide of nickel and hydrogen is thus expressed:—

$$NiO + H = Ni + HO.$$

Here we have at the commencement oxide of nickel and hydrogen; and after the conclusion of the operation there remains metallic nickel and water which has volatilised. If the reducing substance contain both carbon and hydrogen the action will be thus, when a metal (e.g. lead) is reduced from its oxide, carbonic acid and water being formed:—

$$3(PbO) + CH = 3Pb + CO_2 + HO.$$

In the operation of reduction by the aid of carbonaceous matters, two methods are employed: in the one, charcoal, coal, sugar, starch, or any carbonaceous or hydro-carbonaceous body, as argol, is mixed with the substance to be reduced; in the other, the process of cementation is employed. Where sulphides are to be reduced metallic lead or iron is usually employed to remove the sulphur. Generally, however, the sulphides are previously converted into oxides by the operation of roasting, and the reduction is then effected by means of carbonaceous matter.

The process of cementation is conducted by placing the oxide to be reduced in a crucible lined with charcoal, and covering it closely while it is in the furnace; the reduction proceeds gradually from the outside of the oxide to the centre of the mass. The time requisite for this operation depends on three circumstances—viz. the nature of the oxide, the degree of temperature, and the mass acted on.

Some oxides treated this way are reduced very readily; others, again, take a considerable time; while certain of them do not appear to be acted on beyond the outermost layer. Of the first class is oxide of nickel; of the second oxide of manganese; and of the third and last, oxide of chromium.

Each of these classes of reduction has its advantages. The former, or reduction by *mixture* with carbonaceous matter, takes place very quickly and completely, but the reduced metal is often mixed with carbon; in the latter process the residue is comparatively pure, but it is not generally preferred, on account of the time and high temperature necessary.

Reduction by hydrogen gas is very seldom employed; it is, however, necessary in some cases, as for instance in the determination of the percentage of cobalt or nickel in a sample, where perfect accuracy is desirable. The operation is carried on in a tube of hard German glass, having a bulb blown in its centre, which is heated either by a spirit or gas lamp. Attached to it is a tube full of dried chloride of calcium, through which the hydrogen gas effecting the reduction passes to perfectly dry it.

The bulb tube is weighed and the oxide introduced into it; it is again weighed, and the apparatus united by caoutchouc tubes; hydrogen gas (see Reducing Agents) is then passed through it until the whole of the atmospheric air is expelled. Heat is afterwards applied till the bulb is bright red, and the current of gas continued until no more water from the decomposition of the oxide is formed; the source of heat is then removed, and the current of gas continued until the apparatus is cold. The bulb tube, with the reduced metal, is then weighed, and the amount which it has lost represents the oxygen which the hydrogen has removed. By subtracting this oxygen from the original weight of the substance, the difference gives the amount of metal in the amount of oxide operated on.

**Fusion.**—This operation is sufficiently simple, and is employed in all assays by the dry way, in order to obtain, in conjunction with the last process, a button or prill, as it is termed, of the metal whose assay is in progress. It is also a necessary step in the granulation of metals, the preparation of certain fluxes and alloys, also lead for the assay for silver, in order that a homogeneous ingot may be obtained. Some ores, such as those of copper, are melted instead of being roasted or calcined, in order to prepare them for reduction. Minerals are also melted *per se*, or with the addition of borax or carbonate of soda, in order to ascertain the best treatment to be adopted in a subsequent operation. Metals, too, are frequently melted to drive off other volatile metals. In this case the heat should be continued for some time, and should be very high, as it is difficult to remove the last traces of volatile metals. Thus, in melting the spongy gold left behind in the retort after the distillation of gold amalgam, the ingot of gold almost always retains mercury, which can only be removed by repeated meltings at a very high temperature. In some cases the fusion is intended to be only partial, the object being to melt out an easily fusible part of the mineral, for instance, in assaying grey antimony ore, and different bismuth ores.

**Solution.**—In all cases where analysis in the wet way is required, the mineral must be either wholly or partially brought into the state of solution. The choice of a solvent necessarily depends upon the nature of the material under treatment. In some few cases water will be sufficient; but in the majority acids are required. Sometimes advantage will be derived by first extracting all that water will dissolve, and then applying acids to the residue. In speaking of the minerals, &c., which require solution for their assay, the most appropriate solvents will be pointed out. In all cases heat promotes solution.

Solution is best effected in glass flasks; clean Florence oil flasks are very appropriate for most purposes. They may be supported on a hot sand bath, or on a metal ring or coarse wire gauze, over the naked gas or spirit-flame. The flask should be placed in a sloping position, so that when the liquid boils or effervesces from the escape of gas, the drops spirted up may strike against the sloping side, and run back into the liquid instead of being thrown out of the mouth.

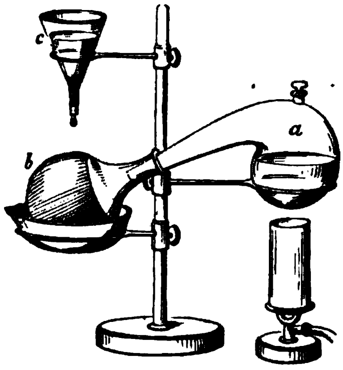
A porcelain dish may also be used, although from the great surface exposed these vessels are more appropriate for evaporation than solution. Beakers may likewise be employed, but they should be covered over with an inverted funnel sufficiently large to rest within the top edge without slipping down more than about half an inch; or a large watch-glass or dial-plate turned concave side upwards may be used as a cover. Both the funnel and dial-plate serve the double object of keeping out dust, and preventing loss of the liquid by projection of fine drops during ebullition.

In many cases solution of the whole or part of a mineral must be preceded by its fusion at a high temperature with carbonate of soda, nitre, or some other flux. The fused mass must then be well extracted by boiling water, when the residue will usually be found soluble in hydrochloric or other acid. Special instructions in this method of effecting solutions will be given in those cases where it is necessary.

**Distillation.**—There are two distinct classes of this operation: in the one, liquids are submitted to experiment, with the object generally of purifying them from substances which are non-volatile, and will consequently be left behind when the liquid comes over. Belonging to this class may be mentioned the distillation of nitric acid, the preparation of distilled water, and the separation of mercury from gold and silver amalgam. In the other kind of distillation, which goes by the name of *dry distillation*, solid bodies, as wood, coal, &c., are subjected to heat in order generally to ascertain the amount of gas or other volatile matter given off in the course of an experiment, from a certain quantity of the coal or other substance operated upon.

In liquid distillations (as in the purification of nitric acid, &c.), retorts are used. The best form for general use is that which is furnished with a stopper at the upper part of the body, *a* (fig. 15), through which the liquid is introduced; the neck of the retort is then placed in that of a receiver, *b*, over which a piece of wet cotton or woollen cloth is placed, and which must be kept cold by means of a stream of water from a funnel, *c*, the shaft of which is partially plugged up with cotton wool. Heat is then applied to the retort, and as much of the liquid as is desired, is distilled over into the receiver. It is advisable not to fill the retort more than two-thirds full, and to apply the heat at first very gently, otherwise there is a risk of breaking the vessel.

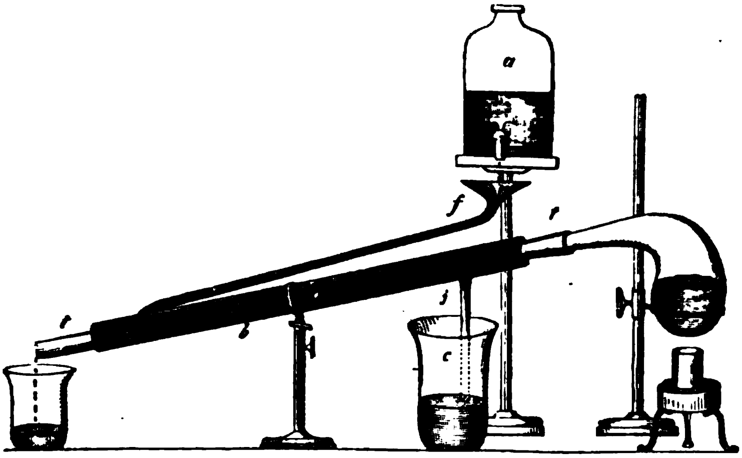
Fig. 15.



A more convenient form of apparatus for distillation and condensation is shown at fig. 16, in which a Liebig's condenser is attached to the retort. Fig. 17 will show the construction of condensing apparatus. The cold water passes into the funnel above, is conveyed at once to the lowest end of the condenser, whilst the heated water passes off by the upper tube.

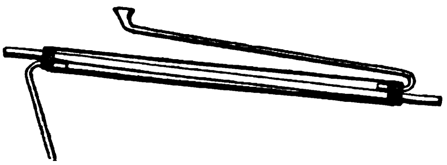
Distilled water is a most important agent in the laboratory; and, as much is needed, it is better to have a still

FIG. 16.



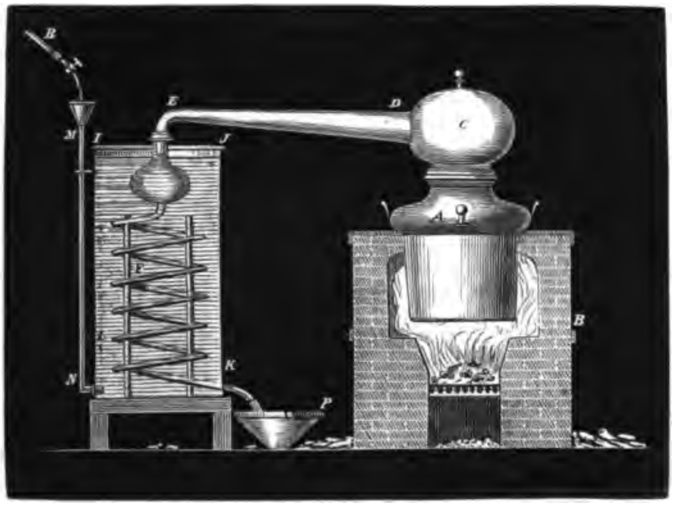
specially adapted for its production. Such a one is depicted at fig. 18, where *A* is the body of the still; *B* the furnace in which it is set (the still may also be placed in the portable furnace, fig. 23, p. 62); *C* the still head; *D E* the neck; *F* the worm; *I J K L* worm-tub containing cold water to condense steam generated in still; *M N* pipe to lead fresh cold water to bottom of worm-tub, while the warm water runs off at the top, as in Liebig's condenser; and *P* the vessel in which the distilled water is received.

FIG. 17.



In the dry distillation of bodies, earthenware, glass, or iron retorts are employed; but for small operations a tube of wrought-iron, about one inch internal diameter, and plugged at one end, is found to be a convenient form of apparatus. It is placed with the substance contained in it in a furnace, and a small tube, either of glass or pewter, is fixed by means of a perforated cork to the open end of the large tube. The gas given off during the operation may be collected by the aid of the pneumatic trough.

FIG. 18.



It will be as well here to describe the pneumatic trough and jars, together with the requisite calculations for temperature, pressure, and moisture to be made in experimenting with gaseous bodies. The pneumatic trough is a vessel of square form, made of tin-plate or zinc, furnished with a shelf at the distance of about three inches from its upper part. This shelf, according to its size, is perforated with one, two, or more holes, each of which is furnished with a small funnel-shaped opening on the inferior side. This opening is for the purpose of receiving the mouth of a gas-delivery tube. The lower part of the trough ought to be furnished with a tap, for the purpose of drawing off the water when it is soiled. The gas jars are made of glass (the most convenient form is cylindrical), and graduated to cubic inches and tenths. Each of the jars may hold from 50 to 100 cubic inches, or more, according to the quantity of gas expected to be furnished during each experiment.

To use the trough, proceed as follows:—Fill it with water to about two inches above the shelf, then fill one of the jars with water; place a ground-glass valve over its orifice, and then set it in an inverted position on the shelf over one of the holes with the funnel-shaped opening, into which introduce the gas-delivering tube. When the mouth of the gas jar is under water, the glass plate is removed. As soon as the gas passes off, by the aid of heat, from the coal or other body in the iron tube, or retort, whichever may have been employed, it will pass into the jar and displace the water. As soon as the jar is full it must be replaced by another, and so on until no more gas passes over The quantity produced in the experiment is then ascertained by reading off the graduations on the jars. It is, however, not the true quantity, as it is in a state of expansion by heat; or it has combined with a quantity of aqueous vapour from the water with which it was in contact; or, lastly, the barometer may not be at the height of 30 inches, from some change in the state of the atmosphere. If it were less than 30 inches it would cause the gas to appear greater in quantity; if more than 30 inches it would appear less in quantity than it really was. The following is the method of making the calculations necessary for reducing the gas to its true volume:—

**Correction for Temperature.**—It has been ascertained by the recent researches of Magnus and Regnault that 100 parts of air or any other gas at 32° of Fahrenheit, when heated to 212°, expand to 136·65 parts, the increase being $\frac{36\cdot65}{100}$ths, or ·3665 of the original bulk. If this be divided by 180, the number of degrees between 32° and 212°, it will be found that air expands $\frac{1}{491\cdot2}$, or in round numbers, $\frac{1}{491}$th of its volume for each degree of Fahrenheit; and we can from this datum determine the expansion or contraction any gas would undergo for any given number of degrees of temperature.

But supposing it be required to know what volume 100 cubic inches of gas at 80° would occupy at 60°, the standard temperature, it must be kept in view that it is not $\frac{1}{491}$th part per degree of the volume at 80° but of the volume at 32°, which is to be deducted; 491 parts of air at 32° become 492 at 33°, and 493 at 34°, and so on; so that at 60° they have increased to 519 parts, and at 80° to 539; so that we have a proportion between the bulks at 60° and at 80°, from which the question may be determined, for:—

| Volume at 80° | | Volume at 60° | | Cubic inches | | Cubic inches |
|---|---|---|---|---|---|---|
| 491 + 48 | : | 491 + 28 | :: | 100 | : | 96·288 |

or the reverse, supposing it were wished to ascertain the real volume at 60° of 100 cubic inches of gas at 40°.

| Volume at 40° | | Volume at 60° | | Cubic inches | | Cubic inches |
|---|---|---|---|---|---|---|
| 491 + 8 | : | 491 + 28 | :: | 100 | : | 104·008 |

**Correction for Pressure.**—As before stated, the standard pressure is 30 inches of mercury, and the law must be kept in mind that the bulk of a body of gas is inversely proportionate to the weight, and directly proportionate to the pressure; so that if we had 100 cubic inches of air when the barometer was 29 inches it would be as:—

30 : 29 :: 100 : 96·6

or if the barometer stood at 31 inches when the 100 cubic inches were measured, it would be as:—

30 : 31 :: 100 : 103·33

so that the rule is:—as the mean pressure is to the observed pressure, so is the observed volume to the true volume. The correction for temperature or pressure may be made indiscriminately, the result being the same in either case.

**Correction for Moisture.**—This correction must be made after the two previous. As before mentioned, the elastic force of the aqueous vapour causes the gas with which it may be mixed to expand, and by reference to tables founded on calculations upon the force of steam at different temperatures, the amount of correction may be easily ascertained. Thus, for 100 cubic inches of a gas saturated with vapour properly corrected to the temperature of 60° and 30 inches pressure, we wish to know the equivalent bulk of the dry gas. The observed volume is partly due to the expansion occasioned by the vapour; and this proportion will be, in proportion to the whole, as the elasticity of the vapour is to the total elasticity; therefore:—

| Elasticity of air | | Elasticity of vapour | | Cubic inches | | Cubic inches |
|---|---|---|---|---|---|---|
| 30·000 | : | 0·560 | :: | 100 | : | 1·86. |

The volume of the dry gas is therefore:—

100 − 1·86 = 98·14 cubic inches.

**Sublimation.**—This operation is a kind of distillation in which the product is obtained under the solid form. The apparatus which may be employed for this purpose are tubes, flasks, capsules, or crucibles. Florence flasks are exceedingly useful: they may be sunk in a sand bath, and the sublimed substance received directly into another flask, or by passing through an intermediate tube. Sometimes, however, it is difficult to entirely remove the sublimed substance; and in order to avoid this inconvenience, Dr. Ure has proposed the following very excellent subliming apparatus:—It consists of two metallic or other vessels, one of which is flatter and larger than the other. The substance to be sublimed is placed in the smaller vessel, and its opening is covered by the larger filled with cold water, which may be replaced from time to time as it becomes hot. The sublimed substance is formed on the lower part of the upper vessel. A large platinum crucible, filled with cold water, and placed on the top of a smaller one, answers the purpose of the before-mentioned apparatus very well.

**Scorification**: **Cupellation.**—These operations will be described under the head of Silver Assay.

# CHAPTER IV.

## PRODUCTION AND APPLICATION OF HEAT.

**Furnaces** for assay purposes may be heated either by solid fuel, oil, or gas, and they may be divided into *wind* and *blast* furnaces. In the former the fire is urged by the ordinary draught of a chimney; and in the latter by means of bellows or artificial blast. We shall commence with the former, as they are in most common use. They are of various kinds, according to the purposes for which they are required. The three principal kinds are those for fusion, calcination, and cupellation. Coal, coke, and charcoal, are the fuels employed, and the merits of each will be particularly discussed. Blast furnaces are only employed for the purpose of fusion, although their forms are various: charcoal and coke are the fuels most in use, but oil and gas blast furnaces are used in small laboratory operations, and for many purposes they are preferable to other furnaces, on account of their freedom from dust and dirt, and the perfect control the operator possesses over the heat.

Furnaces consist of certain essential parts—viz. first, the ash-pit, or part destined to contain the refuse of the combustible employed; secondly, the bars on which the fuel rests; these are sometimes made movable, or fixed to a frame; the former arrangement is more convenient, as it allows clinkers and other refuse matters to be readily removed; thirdly, the body of the furnace in which the heat is produced; and lastly, in wind furnaces, the chimney by which the heated air and gaseous products of combustion are carried off.

**Calcining Furnace.**—Calcining furnaces are small and shallow, because a high temperature is not required. They may be made square or circular; the former are most readily constructed, and where many crucibles are to be heated at once, they are preferable to the circular; but the latter give the greatest degree of heat with the least possible consumption of fuel, and are to be preferred on that account where one crucible only is to be ignited.

The body of the furnace is best made with good bricks, lined with Welsh lump, fire-bricks, or a mixture of Stourbridge clay and sand. It is also desirable that a plate of iron with a ledge be placed over the upper part of the furnace to protect the brickwork from blows with crucible tongs, &c., and to keep it in its place when disturbed by sudden alterations of temperature. The bars of the furnace may be either in one single piece, or made up of several bars of iron fastened to a frame. They ought to be as far as practical from each other, and must not be too large, although large enough not to bend under the weight of the fuel and crucibles, when they become hot, and they must not be so far removed from each other as to allow the coke or charcoal to fall through easily. Lastly, the more readily the air can find access to the centre of the fuel, the higher will be the temperature produced in the furnace: very simple assays occasionally fail, only because the bars are either too large or too close together.

The **Ash-pit** is an open space under the bars, which serves as a receptacle for ashes, clinkers, &c., produced during the time the furnace is in use. It should have the same area as the furnace, and be completely open in front, so that the air may have free access: it is well, however, for the sake of economy, to furnish this opening with a hinged door, having a register plate fixed in it, so that the draught may be reduced, or entirely shut off, in order that the fire may be extinguished when desirable, and fuel saved which otherwise would be burnt in waste.

**Chimney.**—Calcining furnaces generally have no fixed chimney, but are covered with a movable one when a greater degree of heat is required. This chimney may be about five feet high, the diameter of the furnace at the bottom, and tapering off to about two-thirds of that diameter at the top. It is made of strong plate iron, furnished with a wooden handle. The lower part is provided with a door, by means of which the interior of the furnace may be examined without disturbing the whole arrangement of the chimney, and consequent cooling of the contents of the furnace.

If, during the course of any experiment, noxious or offensive vapours are expected to be given off, the furnace must be so arranged that they may be introduced into a flue, by fastening a piece of iron plate pipe, furnished with an elbow-joint, on to the movable chimney before spoken of.

**Evaporating Furnaces.**—The furnaces just described answer exceedingly well in the absence of gas, for heating small flasks, evaporating basins, &c., when surmounted by a tripod stand or sand bath. This is necessary, as many assays by the dry way are preceded or followed by certain operations in the wet way.

**The Hood.**—In order to prevent certain gases or vapours from fires, evaporating basins, &c., from entering into the laboratory, a large metal covering, termed a hood, is employed, terminating in a chimney having a good draught. It is best made of plate or galvanised iron.

**Fusion Furnace: Wind Furnace.**—The wind furnace, properly so called, is a furnace provided with a chimney, and capable of producing a very high temperature.

Wind furnaces are generally square, but if more than four crucibles are to be heated at one time, they may be made rectangular, the chimney being placed at one of the long sides. When the furnace is required to hold but one pot, it may however be made circular.

The body of the furnace ought to be made of good bricks, solidly cemented with clay, and bound by strong iron bands. The bricks must be very refractory, and capable of sustaining changes of temperature without cracking. They are ordinarily made with the clay used in the manufacture of crucibles. In some cases bricks are not used for the lining of this kind of furnace; for instance, a mould of wood is placed in the centre and the open space between the surface of that and the outer brickwork is filled with a paste of very refractory clay, each layer being well beaten down. When the space is filled, the case is withdrawn, and the crust of clay dried with much precaution, every crack that may be caused by unequal dessication being filled up as fast as formed. This method of manufacture is very applicable to circular furnaces. In every case, however, it is necessary to border the edge with a band of iron to prevent injuries from tongs or pots. By using a mixture of 1 part of refractory clay, and 3 to 4 parts of sifted quartz sand, no cracks are formed during desiccation. This mixture is used on the Continent for the interior fittings of Sefström's blast furnace, as well as for larger blast furnaces for manufacturing purposes. It is said to stand a high temperature exceedingly well.

Makins* recommends for small furnaces the second kind of bricks, known as Windsor, or in the trade P. P. bricks. 'These are of a red colour, very siliceous, but soft, easily cut and shaped, and yet standing heat very well. The best method of cutting them is by a piece of zinc roughly notched out as a saw, and then the more accurate figure required may readily be given them by grinding upon a rough flat stone. In this way the small circular furnace formerly made by Newman, and sold by him as his 'universal furnace,' is lined by cutting the bricks with care to the radii of the circle they are to form, when they key in, like an arch, and so need no lining whatever.'

**The Ash-pit.**—On the one hand, it is well to have the power of cutting off access of air into the body of the furnace by the lower part, either to put out the fire entirely, or to deaden it whilst putting in a pot; and, on the other, to attain the maximum of temperature, we must have the means of allowing the air to pass with the greatest possible facility into the furnace. In order to do this it is necessary to furnish the ash-pit with doors, or valves, whereby the quantity admitted may be regulated as desired. It is advantageous to lead the air to the ash-pit from a deep and cold place, by means of a wide pipe. A chimney of less height will then be required.

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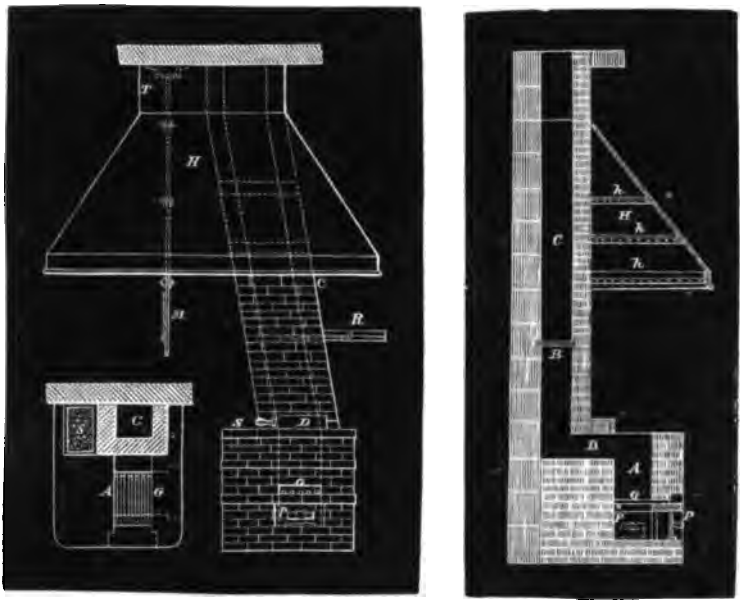
* Makins's Metallurgy, p. 88.

The **Bars** are made in one piece, or are made up of moveable pieces of metal; the latter arrangement is the most convenient. Wherever a wind furnace is in use, the superior opening is closed by a cover made of a fire-tile, encircled with iron.

The **Chimney** is a very essential part of a wind furnace: it is on its height and size that the draught depends, and, in consequence, the degree of heat produced within the furnace. In general, the higher and larger the chimney the stronger is the draught; so that, by giving it a great elevation, exceedingly high temperatures may be obtained.

Fig. 19.



But there is a limit which it is useless to pass in a furnace destined for operations by the dry way; and besides this, the building a very high chimney presents many difficulties, and much expense, so that in laboratory operations, where a very strong current of air is required, recourse is had to a pair of double bellows. A temperature can be produced in a wind furnace sufficiently strong to soften the most refractory crucibles by means of a chimney from thirty-six to forty feet high.

Chimneys are generally made square or rectangular, and have interiorly the same dimensions as the body of the furnace. About two feet above the upper part of the furnace they are furnished with a register or damper, by means of which the current of air may be regulated, or entirely stopped at will. The damper is a plate of iron sliding into a small opening across the chimney.

A wind furnace of the kind above described is represented by fig. 19.

The left hand figure in 19 is the plan, the middle an elevation, and the right is a sectional view. *A* the body of the furnace in which the crucibles to be heated are placed, *G* the bars, and *P* the ash-pit; the cover is formed of a thick fire-tile of the requisite size firmly encircled by a stout iron band, and furnished with a handle for convenience in moving it; *B* the flue, *C* the chimney, *R* the damper; *H* a hood over the furnace, supported by iron bands *h h h*; *M* the handle of a ventilator *T*, which serves to carry off hot air and fumes from furnace when open; and, finally, *S*, a small sand bath, in which to set the red hot crucibles when taken from the fire: one foot square inside the fire-place of the furnace is a very good and convenient size; the remainder will then be in proportion.

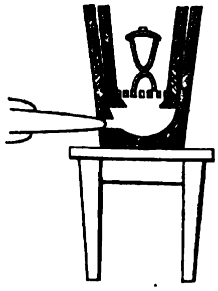
**Blast Furnaces.**—In this species of furnace, the air necessary to keep up the combustion is forced through the fuel by means of a blowing apparatus, instead of being introduced by the draught of a chimney as in the wind furnace.

The most convenient apparatus for forcing air into a furnace is a double bellows; a fan may be used, but it is not so powerful.

The quantity of air passing into a furnace varies with the length of the assay, and ought to increase gradually as the temperature becomes higher.

The following is the description of a most excellent blast furnace which has been in use for some years in the laboratory of the Royal Institution:—The temperature produced by it is extraordinary, considering the small amount of time and fuel employed. It is sufficiently powerful to melt pure iron in a crucible in ten to fifteen minutes, the fire having been previously lighted. It will effect the fusion of rhodium, and even pieces of pure platinum have sunk together into one button in a crucible subjected to its heat.* All kinds of crucibles, including the Cornish and Hessian, soften, fuse, and become frothy in it; and it is the want of vessels which has hitherto put a limit to its application. The exterior (fig. 20) consists of a black-lead pot, eighteen inches in height, and thirteen inches in external diameter at the top; a small blue pot of seven and a half inches external diameter at the top had the lower part cut off, so to as leave an aperture of five inches. This, when put into the larger part, rested upon its lower external edge, the tops of the two being level. The interval between them, which gradually increased from the lower to the upper part, was filled with pulverised glass-blowers' pots, to which enough water had been added to moisten the powder, which was pressed down by sticks, so as to make the whole a compact mass. A round grate was then dropped into the furnace, of such a size that it rested about an inch above the lower edge of the inner pot: the space beneath it, therefore, constituted the air-chamber, and the part above, the body of the furnace. The former was 7½ inches from the grate to the bottom, and the latter 7½ inches from the grate to the top. Finally, a longitudinal hole, conical in form, and 1½ inches in diameter in the exterior, was cut through the outer pot, forming an opening in the air-chamber at the lower part, its use being to receive the nozzle of the bellows by which the draught was thrown in.

Fig. 20.



Sefström's Blast Furnace, obtainable at most chemical instrument makers, is also very powerful and convenient;

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* Faraday.

it consists of a double furnace. It is made of stout sheet iron, lined with fire-clay, and is used with coke, or charcoal and coke, cut into pieces of about a cubic inch in size. The blast of air is supplied by a powerful blowing-machine It will readily produce a white heat. Indeed the limit to its power seems to be the difficulty of finding crucibles or interior furnace fittings which will stand the temperatures produced in it without softening. Kersten states that he increases the heat in Sefström's blast furnace by using a hot blast.

H. Ste-Claire Deville has employed for melting platinum a furnace of 30 centim. height, and 28 centim. wide, which rests upon a cast-iron plate full of holes. This is connected with a forge bellows. After blowing for a few minutes, the temperature of the furnace will have reached the highest possible degree, but this zone of maximum heat only extends to a small height above the bottom of the furnace. Above this point a considerable quantity of carbonic oxide gas is formed, which burns with a very long flame. The heat produced in this furnace is so high, that the best crucibles melt, and only crucibles made of good and well burned lime can be used.

The **Muffle**, or **Cupel Furnace**, is a furnace in the centre of which is placed a small semi-cylindrical oven, which is termed the muffle. These furnaces were in use as early as the thirteenth century. Their construction and dimensions depend:—

1. On the temperature which the furnace is intended to produce;
2. On the number of cupellations required to be performed at one time; and
3. On the kind of fuel used.

The muffles are mostly made of refractory clay, generally in one piece, but it is best to make them in two pieces; the bottom being one, and the cover or vault the other.

Muffles are sometimes made of cast iron—cast in one piece. They are useful in small furnaces intended for cupellations only.

Muffle furnaces must always be provided with a good chimney to carry off the hot gases.

The muffle, being completely surrounded by ignited fuel, acquires a very high temperature, and in its interior all operations requiring the presence of air, and which cannot be carried on in contact with carbonaceous matters, may be performed—such as roastings, scorifications, and cupellations.

When from ten to twenty cupellations have to be effected at one time, large brick furnaces are employed; and, in consequence, much fuel is consumed to waste in these when only a few cupellations are required. This has occasioned many persons to endeavour to form small furnaces, where one or two cupellations may be carried on with the smallest possible quantity of fuel.

MM. Aufryé and d'Arcet have contrived a furnace which is capable of fulfilling all these conditions.

The furnace is elliptical, and about 7 inches wide and 18 high; its ash-pit has but one circular opening, and its height is such, that when the furnace is placed upon it, and the whole upon a table, the assayer can, when seated, readily observe the course of the assay within the muffle. The hearth has five openings, in one of which the muffle is placed; in another, a brick to support it; a third is for the purpose of introducing a poker to stir the ashes, and make them fall through the grate-holes: this can be closed with a small earthen plug; and lastly there are two round holes, placed in its largest diameter, to facilitate the introduction of air, either by draught or a pair of bellows, as the case may require. The support for the fuel is generally a plate of earthenware, pierced with holes, and bound round with iron wire to keep it together in case it cracks by changes of temperature; but it is better to use an iron grating.

The dome of the furnace has a circular opening, which can be closed by a plug of earthenware: this opening serves for the introduction of the fuel. A chimney is necessary to increase the draught; it is made of sheet iron, and may be from 1½ to 2 feet in height, and ought to fit the upper part of the dome very exactly. At its base there is a small gallery, also of sheet iron, in which it is intended to place the new cupels, so that they may be strongly heated before introduction to the muffle. This saves many of them from fracture.

M.M. Aufryé and d'Arcet have estimated the quantity of charcoal necessary to heat this furnace. The following are comparative experiments:—

| No. | Silver employed, grains | Lead employed, grains | Time of assay, minutes | Standard thousandths | Charcoal used, grains |
|---|---|---|---|---|---|
| 1 | 1 | 4 | 12 | 947 | 173 |
| 2 | 1 | 4 | 11 | 950 | 86 |
| 3 | 1 | 4 | 13 | 949 | 93 |
| 4 | 1 | 4 | 10 | 949 | 60 |

Coke or charcoal may be used in this furnace, but the fire must be lighted by means of charcoal alone, as coke is very difficult to inflame in a cold furnace. When it is red-hot, it may be fed with coke, or, better still, a mixture of coke and charcoal.

Where great numbers of cupellations have to be made at once, the following form of brick furnace is requisite.

Fig. 21 shows an elevation of the furnace; fig. 22 shows a section. The interior of the furnace is of fire-brick; the exterior of common brick. The upper part is protected by a plate of iron, and the superior opening, through which the fuel is introduced, is covered when necessary by a large fire-tile strongly encircled with an iron band, to which are attached two handles, by which the whole can be moved.

The muffle opening as seen partially open in the diagram, can be entirely closed by means of two sliding doors, made of sheet-iron, running in a stout wrought-iron frame, built into the brickwork. Immediately below the muffle entrance are two movable bricks; these close the openings through which the fire bars are introduced; and still lower down is the ash-pit door, furnished with a register for the better regulation of the current of air required by the furnace. In fig. 22 is shown a brick built into the back of the furnace, on which the close end of the muffle is supported. This brick may, however, be replaced by a crucible or fire brick standing on the bars of the furnace.

A very useful furnace for small operations is one which has been aptly termed the 'universal furnace,' as it is capable of performing all that is required of any furnace in an assay (see figs. 23 and 24, elevation and section). It is to be much recommended for its durability, cheapness, and its small size compared with the heat it can produce. It is constructed

FIG. 21. FIG. 22.

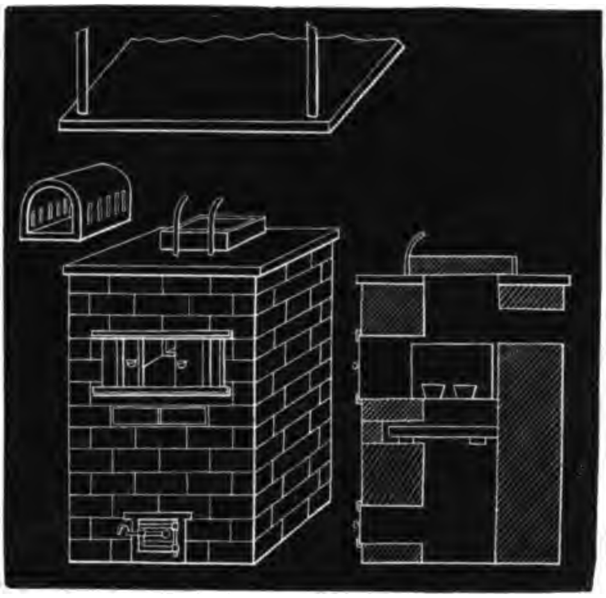
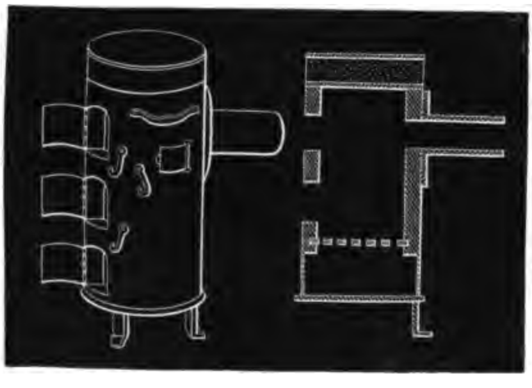


FIG. 23. FIG. 24.



externally of sheet iron, very stout, and is lined with fire-brick, not cemented together, but ground and keyed as an arch, so that it can never fall out till it is completely useless. Its height is about 2¼ feet and diameter 1 foot; internal diameter 8 inches and depth of fire-place 1¼ feet. It is furnished with five doors, one in the ash-pit and four in the body of the furnace, two in the front, one above the other, and two opposite each other, at the sides. The cover serves as a sand-bath, and when that is taken off there is a series of cast-iron rings, fitting the top of the furnace, for the purpose of placing basins either for the purpose of evaporation, calcination, or roasting. The two opposite holes serve for the introduction of a tube in operations where it is requisite to pass a gas over any body at a red heat. In the lower hole in front, can be placed a muffle for roastings and cupellations, introducing fuel and crucibles by the upper one; it also serves as an opening through which the state of the furnace can be seen, or the progress of any assay observed.

Iron, manganese, nickel, and cobalt, can be fused in this furnace, when it has a flue of about thirty feet in height attached to it, and by closing the ash-pit door, the dullest red heat, for gentle roastings, can be obtained.

## FURNACE OPERATIONS.

Crucibles must be carefully supported in the fire, and must always be covered. They must stand solidly, and be at equal distances from the sides and bottom of the furnace, so as to receive a like share of heat, and they must be completely surrounded with the fuel. If a crucible is supported on the grate bars of a furnace the draught of cold air will prevent the bottom from getting hot. If it is supported on the fuel, the bottom gets heated quickly, but the fuel in burning away allows the crucible to fall down, and may cause the loss of the contents. For these reasons a crucible should always be supported on a piece of fire-brick about three or four inches high. In many cases an old crucible inverted will serve as a convenient support. The fire must be got up gradually, so as to prevent the sides of the furnace and the crucibles within from cracking from the sudden increase of heat. No time is saved by urging the fire strongly at first, for crucibles are bad conductors of heat, and a high temperature at the commencement scarcely causes the heat to penetrate to the interior faster than a moderate redness. After the furnace has arrived at a full red heat, more air may be given, and in from about twenty minutes to one hour, the assay will be finished. During the time the furnace is in full action, the cover must be occasionally removed to add more fuel, if any open spaces occur round the crucibles; also to press the fuel close to the pots. When the pots are taken out they may be placed on the anvil or in a sand-bath, and allowed to cool gradually, before they are broken to examine their contents.

In commencing a second assay immediately in the same furnace, certain precautions must be taken to insure success. In the first place, all ash and clinker must be removed from the grate, by means of a crooked poker; secondly, the fuel must be pressed down firmly; and lastly, a layer of fresh combustible must be placed on the fire, and before that is ignited the crucibles must be arranged upon the support and the spaces about them be filled with coke or charcoal, as the case may be, and the assay be proceeded with as before.

In executing many assays, one after the other, a great saving of fuel is effected, for the furnace is not allowed time to cool.

**Auxiliary Apparatus.**—Ordinary assay furnaces require very few instruments; they are, firstly, pokers or stirring rods, made of stout bar-iron: these may be straight, as for stirring the fuel from the top of the furnace, so as to fill up cavities formed by uneven combustion; or curved, for clearing the bars from below from clinkers and ashes. Straight and curved tongs are also required; for small crucibles the blacksmith's common forge tongs are the most suitable; tongs with semicircular ends (see fig. 25) are very serviceable for larger crucibles. The tongs *a*, are particularly adapted for removing large cupels or calcining tests from the muffle; the tongs *b* and *c* are used for lifting heavy crucibles from the wind or blast furnace. In case the eyes of the operator are weak, it is advisable to make use of a pair of deep neutral-tint spectacles. Most of the radiant heat from the interior of a furnace may be cut off by holding before the face a large sheet of window glass; or the operator may look at the reflected image in a looking-glass instead of looking direct into the furnace itself. Some assayers recommend the use of masks for the face and gloves for the hands; but these are not needed. In cupel furnaces, both curved and straight pokers or stirring rods are required; also a curved rod made of lighter iron, to be used in closing the sliding doors, slightly moving cupels, &c. The tongs used vary in form (see fig. 26). *a* represents very light elastic tongs or pincers employed in the introduction of lead and other matters to the cupel: *b* shows the tongs for holding the scorifier; the curved part fits the lower part of the scorifier, and the upper or single part passes over the upper part of the scorifier, so that its contents may be emptied into the proper mould without fear of its slipping from the operator's grasp: *c* represents the tongs used in moving cupels; they are slightly curved, so that the cupels from the back part of the muffle

Fig. 25.

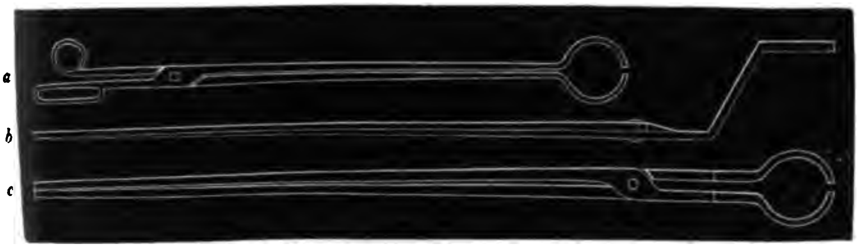
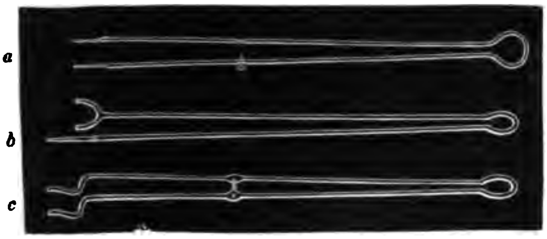
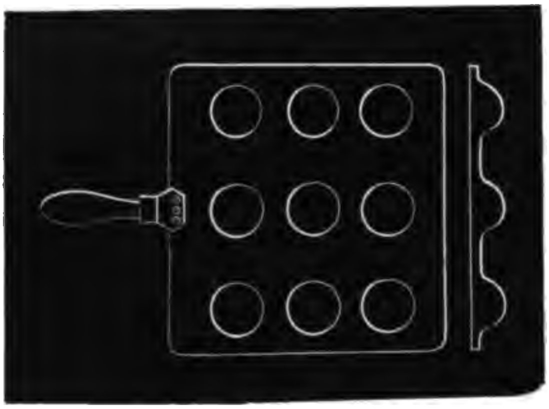


Fig. 26.



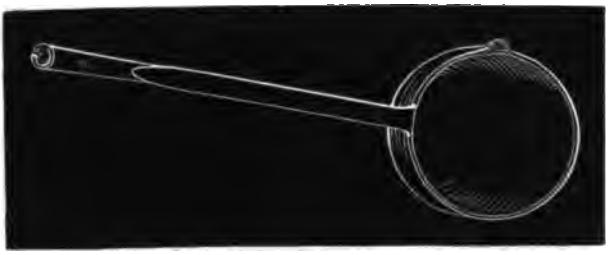
may be removed without disturbing those in front. Fig. 27 shows the plan and section of the ingot mould, into

FIG. 27.



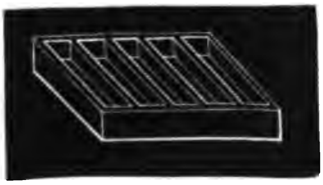
which the contents of the scorifiers are poured: it is made of thin sheet iron, and the depressions for the reception of the fused lead, slag, and ore are hammered out. Fig.

FIG. 28.



28 is a wrought-iron ladle, in which lead clippings, &c., are melted, in order to obtain a fair average of a large quantity; and fig. 29 represents the ingot mould into which the fused lead, or other metal, is poured. Other special apparatus will be described under the assay in which they are required.

FIG. 29.



Furnaces are heated with anthracite, coke, and charcoal, and sometimes with a mixture of the two latter; coal is very seldom employed, and therefore will not be much spoken of; coke is the principal combustible used in assaying. Calcining furnaces ought to be heated with charcoal alone, because coke employed in small quantities ignites and burns with too much difficulty. All fuel contains certain fixed matters which remain after combustion, and which constitute the ash. This ash fuses or agglutinates together, and when a certain quantity is formed, if it be not removed, the fire will decrease in intensity, and finally die out. As all combustibles do not contain the same amount of ash, they should be carefully selected; those containing the least are to be preferred; in the first place, because, weight for weight, they contain more available fuel; and secondly, because they can be used in a furnace a longer time without the formation of so much clinker. The composition of the ash likewise merits much attention.

Charcoal contains, in general, from 3 to 4 per cent. of ash, the chief components of which are carbonates, lime and potash. Certain other matters are also present, as phosphoric acid, oxide of iron, manganese, &c., but these are in very minute proportions. The ash is not fusible *per se*, and if it does not meet with any substance capable of combining with it, it passes through the bars as a white powder; but when the potash predominates, it exercises a corrosive action on the bricks with which the furnace is lined, as also on crucibles, lutes, &c., by the formation of a fusible silicate of potash, which in course of time runs down the sides of the furnace, and chokes the bars. Whenever the ash is in very small proportion to the charcoal, its fusion is rather useful than otherwise, because it forms a species of varnish, which, penetrating the surface of the bricks and lutes, gives them solidity by binding them together with a cement, forming part of their substance.

The proportion of ash which coke contains is very variable; some commercial samples contain from 8 to 10 per cent., while others made from very pure coal, give but 2 to 3 per cent.; so that this fuel also ought to be carefully chosen. The nature of this ash is different from that of charcoal; it consists principally of oxide of iron and clay. The former is produced from the pyrites which coal generally contains. The clay is similar to the carbonaceous schists, not very fusible by itself, but nevertheless capable of softening. When pure, it forms a slag, which attacks neither the bricks nor crucibles. This happens very rarely; it is more often that oxide of iron predominates, and this by contact with the carbonaceous matter, becomes reduced to the state of protoxide, and is then not only very fusible, but exercises on all argillaceous matters a very corrosive action, so that crucibles are very seriously injured, and the sides of the furnace require frequent repairs.

Weight for weight, coke and charcoal give out nearly the same quantity of heat; but in equal bulks, the former developes much more heat, because its density is greater: from this difference in the calorific power of coke and charcoal, it results that in the same furnace the former produces a greater degree of heat than the latter; and at high temperatures the difference has been proved to be nearly 10 per cent. In order to account for this, we must consider, firstly, that in a given space the quantity of heat produced in a certain time (and, in consequence, the temperature) depends on the amount of fuel burnt, and increases with its weight; secondly, that combustion takes place but at the surface of the masses, whatever may be the nature of the fuel; from which may be deduced, that the weight of fuel burnt in an unit of time ought to be exactly proportionate to its density; and, in consequence, the densest fuels, furnishing the most food for combustion, ought to give out the greatest heat. But, as for the same reason they consume a larger proportion of oxygen, they require, in order to produce the maximum effect, a more rapid and stronger current of air.

It is clear from what has been stated concerning the relative properties of coke and charcoal, than when the former can be procured of good quality, and especially when the ash contains but little oxide of iron, it ought to be preferred to charcoal, for assays requiring a high temperature.

This being an important subject, it has been thought advisable in this edition to devote a special chapter to the assay of fuel. (See chapter v. p. 133.)

A very essential condition in obtaining the maximum effect of a furnace, the importance of which can alone be appreciated by experience, is to choose pieces of fuel of a suitable size. If, on the one hand, a shovelful of coke or charcoal be taken at random, it generally contains the dust and dirt found in most fuel, and which, by filling the interstices, prevents the air from passing as required, and consequently renders the combustion slow. On the other hand, if a furnace be filled with large pieces, considerable spaces are left between them, so that but a comparatively small surface is exposed to the action of the atmospheric oxygen, and a correspondingly small quantity of fuel is consumed in a given time; so that the maximum heat can never be obtained. In order to produce the desired result, it is necessary that the pieces shall have a certain mean size, and experience has proved that pieces about 1 inch to 1¼ inches diameter produce the best effect. These may be selected by sifting the coke through two strong wire sieves, one of which has meshes about 1¼ inches square, and the other about 1 inch square. The coke which passes through the larger one, but will not go through the smaller sieve, will be the right size for the furnace.

**The Effects produced by Wind and Blast Furnaces.**—Assays by the dry way can be made either in wind or blast furnaces. In either of them, the degree of heat depends upon the volume of air which passes through the fuel in the same time; but, *cæteris paribus*, large furnaces produce more heat than small ones, because comparatively less heat is lost by radiation in the former than the latter.

In a wind furnace, the maximum of heat is limited by the size of the chimney, and in a blast furnace, by the dimensions of the bellows; but by weighting the latter, more or less, the force of the blast can be increased, and, in consequence, the temperature to a considerable extent. In this respect blast have the advantage over wind furnaces.

In the latter, the draught increases in proportion as the heat becomes more intense in the furnace, so that the temperature producible increases progressively. In a blast furnace, the bellows can be weighted and worked as heavily as possible at once, and by opening all the apertures for receiving air, the maximum temperature can be produced *more rapidly* than in a wind furnace; but this is of little use, because as heat passes very slowly through the substance of a crucible, when the object is to fuse its contents it must be heated gradually, so as to avoid running the risk of softening the crucible before its contents are acted upon, or even scarcely made warm.

Wind furnaces are, however, much more serviceable and economical than blast, because they work themselves, and do not require the service of a man to attend to the bellows. A blast furnace is used in a laboratory, in certain cases; for instance, when a single crucible has to be submitted to an intense heat, and when the furnace is small, and the bellows large, in which case the operation resembles a blow-pipe assay.

In whatever manner the air is introduced into any kind of furnace, either wind or blast, it is evident that the quantity of heat developed in equal-sized furnaces depends upon the quantity of air introduced in the same time: but the degree of temperature is not the same in different parts of the furnace, and the distribution of heat varies according to the manner in which the air is introduced into the midst of the fuel. The side over which the air passes, is kept cold by the current, on which account fire-bars last a long time without becoming oxidised, but the heat rapidly augments up to a certain distance from the bars, at which place it arrives at its maximum; above that it diminishes rapidly, because the air is nearly deprived of its oxygen. Experiment has proved that this maximum is about 2½ to 3 inches above the bars or tuyeres.

In common wind furnaces the air enters through the spaces between the horizontal bars which form the bottom of the furnace, and the crucibles are placed on a stand which rests on these bars. By this means the lower and centre part of the crucibles, in which parts the matter to be fused is placed, are exactly situated in the maximum of heat, but the stand being constantly kept cold, by the contact of a current of air, establishes a continual draining or carrying away of heat from the interior of the crucible outwards, so that the substance submitted to assay can only arrive at the maximum temperature after a length of time, and the maximum then is always inferior to that in the mass of fuel. It is on this account that assays in a blast or wind furnace generally occupy from one hour to two hours. The author has found that the time may be reduced to half that just stated, if a good solid foundation of fuel be made, and the crucible placed on that, and well surrounded by coke, constantly kept close to the pot and the sides of the furnace: in this manner the cooling effect of the stand is removed, and the consequent maximum effect of the furnace produced, but then there is danger of the supporting fuel being burnt away from the crucible and the latter getting upset.

## OIL AND GAS BLAST FURNACES.

It sometimes happens that metallurgists and assayers have occasion to melt metals at a white heat, but do not wish to heat a large furnace for the purpose. In these cases either the gas or oil furnaces, now to be described, will prove very useful.

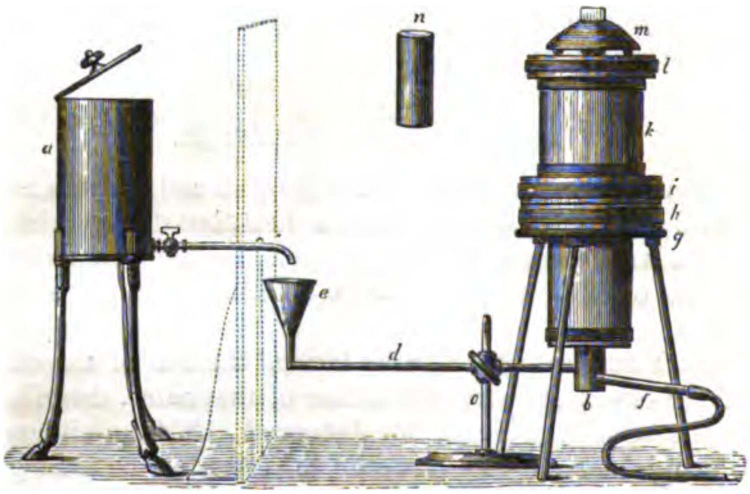
**Oil Furnaces.**—Mr. Charles Griffin, the son of the well-known chemical instrument maker of that name, described, in the 'Chemical News,' for January 2, 1864, an oil lamp, which is not only as powerful in action as the best gas furnaces, but almost rivals them in handiness and economy.

**Description of the Apparatus.**—The oil-lamp furnace is represented in perspective by fig. 30, and in section by fig. 31. It consists of a wick-holder, an oil-reservoir, and a fire-clay furnace; to these must be added a blowing-machine for the supply of atmospheric air.

The oil-reservoir is represented at *a*, fig. 30; it is made of japanned tin-plate, mounted on iron legs, and fitted with a brass stop-cock and delivery-tube. Its capacity is a little more than a quart. The wick-holder is represented at *b*, and the upper surface of it by the separate figure *c*, fig. 32. The wick-holder and the oil-reservoir are consequently detached. *d* is a tube which brings oil from the funnel *e*, and *f* is a tube to be placed in connection with the blowing apparatus. The wick-holder contains three concentric wicks, placed round the multiple blowpipe *c*, which is in communication with the blowing tube.

The crucible furnace consists of the following parts, shown in figs. 30 and 31 :—*g* is an iron tripod; *h* is a flue for collecting and directing the flame. This flue is of such a width, that when the wick-holder, *b*, is pushed up into it until the top of the wick is level with the top of the clay cone, there remains a clear air-space of about ⅛ inch all round between the wick-holder and the cylindrical walls of the flue. *i* represents a fire-clay grate having three tongues, shown by *i* (fig. 32), on its upper surface. These tongues support the crucible, without stopping the rising flame. *k* is a fire-clay cylinder which rests upon the grate *i*, and encloses the crucible, forming, in fact, the body of the furnace. Of this piece there are three sizes : the smallest is of 3 inches bore, and works with crucibles that do not exceed 2¾ inches diameter ; a middle size, 4 inches bore, for crucibles not exceeding 3¾ inches diameter ; the largest size, 5 inches bore, for crucibles not exceeding 4¾ inches

Fig. 30.



diameter. This piece being heavy, is provided with handles, as represented in *p*, fig. 32. The walls of the cylinders are from 1 inch to 1½ inch thick. *l* is a flat plate of fire-clay with a hole in the centre, used to cover the cylinder *k*, so as to act like a reverberatory dome; *m* is a cover which prevents loss of heat from the crucible by radiation, but gives egress to the gaseous products of the combustion of the oil; *n* is an extinguisher to put over the wick-holder when an operation is ended; and *o* is a support for the wick-holder. No chimney is required.

**Management of the Oil-lamp Furnace.**—The apparatus is to be arranged for use as it is represented by fig. 30. The cylinder, *k*, is to be selected to fit the crucibles, and the crucible of a size to suit the quantity of metal that is to be melted: 1 lb. of iron requires the smallest of the three cylinders, described above; 1½ lb. the middle size; 5 lbs. the largest size. The air-way between the crucible and the inner walls of the cylinder should never exceed ¼ inch nor be less than ⅛ inch.

Fig. 31.

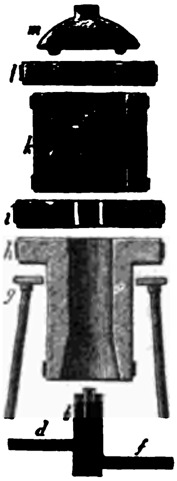
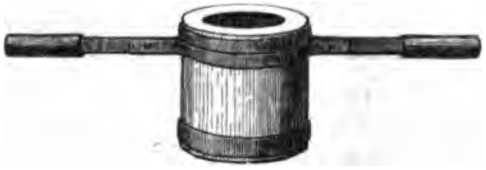
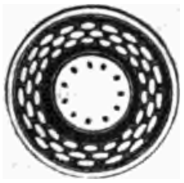
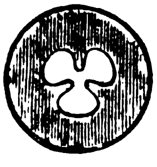


Fig. 32.







The cotton wicks must be clean, and be trimmed a little below the level of the blowpipe *c*. If properly managed, they do not readily burn away, but can be used for several fusions. The reservoir should be filled with oil for each operation. The proper sort of oil for use is the more volatile kind of mineral oil, of the specific gravity of 750, which is now easily procurable at about three shillings per gallon. The variety known by the commercial name of turpenzine answers well. The combustion of a quart of this oil, costing ninepence, gives heat sufficient to melt 5 lbs. of cast iron. Probably the lighter kinds of paraffin oil may be suitable. Liquids of the alcohol class, spirits of wine, and pyroxylic spirit can be used; but they are less effective and more expensive than turpenzine. Care must be taken not to spill the oil on the table or floor, and not to decant it carelessly in the neighbourhood of a light, because atmospheric air strongly charged with the vapour of these light oils is explosive. When the oil is burnt in the furnace in the manner described below, there is no danger. During an operation, a wooden screen, as represented by the dotted lines in fig. 30, should be placed between the oil-reservoir and the furnace, to prevent the vaporisation of the oil by radiant heat. As the wick-holder *b*, and supply pipe *d*, contain only about one fluid ounce of oil, the oil must run continuously during a fusion, from the reservoir *a* into the funnel *e*, in order that the cotton may be always flooded. The success of the fusion depends upon the due supply of oil, to which point the operator must pay attention. At the commencement of a fusion, the oil must be run from the reservoir until the surface of the oil in the funnel has a diameter of about an inch. The wicks will then be flooded, and a light may be applied, and a gentle blast of air then set on. The oil immediately sinks in the funnel, and the stop-cock must be opened and so regulated as to keep the oil barely visible at the bottom of the funnel. If too much oil is supplied it immediately rises in the funnel, and simultaneously overflows the wick-holder. Too much vapour is then thrown into the furnace, and the heat is immediately lowered, especially at the beginning of an operation, before the fire-clay portions of the furnace are well heated. If, on the contrary, too little oil is supplied, the wicks burn, and the operation is spoilt. The demand of the wick-holder for oil depends upon the condition of the furnace and the character of the fusion in progress. When the lamp is newly lighted and the furnace cold, the oil should be passed slowly in distinct drops; but as the furnace becomes heated the rapidity of the supply of drops should be increased; and finally, when the furnace is at a white heat, the oil should be supplied in a thin continuous stream. When the fusion to be effected is that of only a small quantity of metal, such as 1 lb. of iron, a rapid supply of drops of oil is sufficient even to the close of the operation. At that rate the burner consumes about 1¼ pint of oil in an hour. When the fusion to be effected is that 4 lbs. or 5 lbs. of iron and the large furnace is in action and has been brought to a white heat, the supply of oil must, as stated above, be in a thin continuous stream, and the operation will then consume two pints of oil in the hour. And here it requires remark that, with that continuous supply, when the furnace is large and is at a white heat, the oil does not rise in the funnel, being instantaneously converted into gas at the mouth of the burner, and thrown up in that state into the furnace for combustion. The operation, indeed, consists at that point of a rapid distillation of oil-gas, which is immediately burnt, in the presence of air supplied at a suitable pressure by a dozen blowpipes, in effective contact with the crucible to be heated.

The flame produced in this furnace is as clear as that produced by an explosive mixture of air and coal-gas. It is perfectly free from smoke, and the consumed vapours which occasionally escape with gaseous products of the combustion, are even less unpleasant to smell and to breathe in than are those which are usually disengaged by a blast gas furnace, or by an ordinary lamp, fed with pyroxylic spirit.

The contents of a crucible under ignition in this furnace can at any moment be readily examined, it being only necessary to remove the pieces *l* and *m* with tongs, and to lift the cover of the crucible, during which the action of the furnace need not be interrupted.

When the operation is finished, the blast is stopped, the stop-cock is turned off, the oil-reservoir is removed, the wick-holder is lowered on the support *o*, withdrawn from the furnace, and covered with the extinguisher *n*. The quantity of oil which then remains in the lamp is about one fluid ounce.

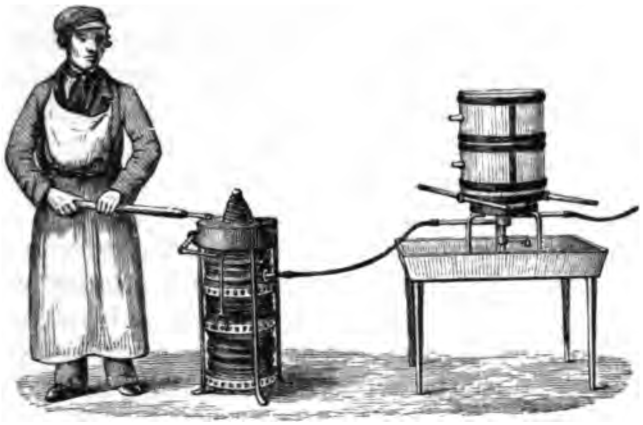
**Power of the Oil-lamp Furnace.**—The furnace being cold when an operation is commenced, it will melt 1 lb. of cast iron in 25 minutes, 1½ lb. in 30 minutes, 4 lbs. in 45 minutes, and 5 lbs. in 60 minutes. When the furnace is hot, such fusions can be effected in much less time; for example, 1 lb. of iron in 15 minutes. It need scarcely be added, that small quantities of gold, silver, copper, brass, German silver, &c., can be melted with great ease, and that all the metallurgical and chemical processes that are commonly effected in platinum and porcelain crucibles can be promptly accomplished in the smallest cylinder of this furnace; and in the case of platinum vessels, with this special advantage, that the oil-gas is free from those sulphurous compounds the presence of which in coal-gas frequently causes damage to the crucibles.

**Requisite Blowing Power.**—The size of the blowing-machine required to develop the fusing power of this oil-lamp furnace depends upon the amount of heat required or the weight of metal that is to be fused. For ordinary chemical operations with platinum and porcelain crucibles, and even for the fusion of 1 lb. of cast iron in clay or plumbago crucibles, a blowing power equal to that of a glass-blower's table is sufficient, provided the blast it gives is uniform and constant. But the fusion of masses of iron weighing 4 or 5 lbs. can be effected by the gas which this oil-lamp is capable of supplying, provided a sufficiently powerful blowing-machine supplies the requisite quantity of air. When more than a quart of oil is to be rapidly distilled into gas, and the whole of that gas is to be instantly burned with oxygen, it is evident that effective work demands a large and prompt supply of air.

As in all practical matters of this sort, the *cost* is a main question, it may be useful to state that the price of this apparatus complete, without the blowing-machine, but including every other portion necessary for heating crucibles up to the size sufficient to fuse 1 lb. of cast iron, is one guinea; and that, with the extra furnace pieces for crucibles suitable for 5 lbs. of iron, or any intermediate quantity, the cost is one guinea and a half.

**Gas Blast Furnace.**—The furnace (shown at fig. 33) is suitable for the fusion of refractory metals, and for all purposes of ignition, combustion, fusion, or dry distillation at a red or a white heat, where it is desirable to produce those temperatures and effects promptly, steadily, and conveniently. This furnace has also been devised by Mr. Griffin.

Fig. 33.



It consists of two parts: firstly, of a particular form of gas-burner, which is supplied with gas at the usual pressure, and with a blast of common air, supplied with bellows or a blowing-machine, at about ten times the pressure at which the gas is supplied; secondly, of a furnace which is built up round the flame that is produced by the gas-burner, and the crucible that is exposed to ignition. The object of the peculiar construction of this furnace is to accumulate and concentrate to a focus the heat produced by the gas-flame, and to make it expend its entire power upon any object placed in that focus.

Fig. 34.

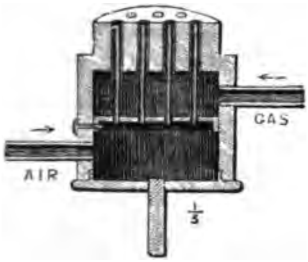
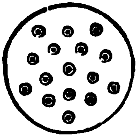


Fig. 35.



The gas-burner is a cylindrical iron reservoir, constructed as shown in fig. 34, which is drawn on a scale of one-third the full size. It contains two chambers, which are not in communication with one another. Into the upper chamber, gas at ordinary pressure is allowed to pass by the tube marked GAS. Into the lower chamber, air is forced by the tube marked AIR. The upper part of the burner is an inch thick in the metal. Through this solid roof holes are bored for the escape of the gas. The experiments described hereafter were chiefly made with a burner that contained sixteen holes, arranged as shown in fig. 35, which is a surface view of the burner represented by fig. 34. But burners with three holes, six holes, and twenty-six holes, have been made for other purposes. The number of holes depends, of course, upon the heating power required from the burners. The air passes from the lower chamber, through a series of metal tubes placed in the centre of the gas-holes, and continued to the surface of the burner, so that the gas and air do not mix until both have left the gas-burner, and then a current of air is blown through the middle of each jet of gas. The bottom of the gas-burner is made to unscrew, and the division between the two chambers which carries the air-tubes is easily removable for the purpose of being cleaned. The gas and air pipes are both half an inch in the bore, and may be about ten inches long; the gas should have a pressure of half an inch of water, and the blast of air about ten times that pressure. The quantity of gas used in an hour is about 100 cubic feet. The stop-cock which supplies it has a bore of half an inch. The round rod which is represented at the bottom of the burner, fig. 34, is intended to fit it to the support, shown by *b*, in figs. 36 and 41.

When the gas is lighted and the blast of air is put on, the flame produced by the gas-burner is quite blue and free from smoke. It is two inches in diameter, and three inches high, and the point of greatest heat is about two inches above the flat face of the gas-burner. Above this steady blue flame there rises a flickering ragged flame, several inches in height, varying with the pressure of the gas. In the blue flame thin platinum wires fuse readily.

When the gas is burning in this manner, and the apparatus is attached to flexible tubes, the burner may be inverted or held sideways, without disturbing the force or regularity of the flame, so that the flame may be directed into a furnace at the bottom, the top, or the side, as circumstances may require.

The following articles are used in building up the gas furnace for different experiments. They vary in size according to the volume of the crucible, or the weight of the metal which is to be heated.

A circular plate of fire-clay, two inches thick, with a hole in the centre, exactly filling the upper part of the gas-burner, which is made to enter into the hole three-quarters of an inch. In external diameter this clay plate agrees with each size of furnace.

A cylinder of fire-clay, of which two pieces are required to constitute the body of each furnace. In the middle of each cylinder a trial-hole is made, one inch in diameter, to which a fire-clay stopper is adapted. (See fig. 36.)

A fire-clay cylinder, closed at one end, and pierced at the open end with numerous holes of half an inch in diameter. The thickness of the clay is immaterial. There are several sizes of this cylinder for crucibles of different diameters. It is represented at *a*, fig. 37.

A circular plate of fire-clay from two to four inches in diameter, and one inch thick. Similar pieces half-inch thick are useful.

Cylinders of plumbago, pierced with numerous holes of three-eighths of an inch in diameter. Their use is to support flanged crucibles over the flame.

A cover or thin plate of plumbago, three inches in diameter. It has a small hole in the middle, and being of a soft material, the hole can be easily cut or filed to suit crucibles of any desired size, so as to support them on the cylinder.

As in all cases the heating power of the gas furnace spreads laterally, and does not rise vertically, the most advisable form of the crucibles required for use in it is *short and broad*, not tall and narrow, and the supporting cylinders must be shaped accordingly. No fire-bars or grates can be used to support crucibles in this gas-furnace, because no material, formed into narrow bars, can sufficiently withstand its powers of fusion and combustion.

A plumbago cylinder, or crucible-jacket, two and a half inches high, two and a-half inches in diameter, and a quarter of an inch thick in the walls. It has several holes of three-eighths of an inch in diameter.

A circular cover or dome, flanged at the bottom, and having a knob or handle at the top. It is pierced with twenty-four holes of a quarter of an inch in diameter, arranged in two rows near the bottom. This dome, when of small size, is made of plumbago; when of large size, of fire-clay.

Plumbago crucibles made with a flange or solid overhanging rim, the use of which is to suspend the crucibles over the gas-burner, by means of the cylinders. When the crucibles are too small to fit the cylinders, a flat plate is filed to fit the crucible, and is then placed on the cylinder, to the diameter of which it is adapted.

Besides these pieces of fire-clay and plumbago, it is necessary to be provided with a strong iron tripod, to sustain the furnace, as represented by *c* in fig. 36, an iron pan in which to place the furnace, and a quantity of gravel or rounded flints, not less than half an inch, nor more than one inch in diameter. These pebbles form an essential part of this gas furnace.

**Gas Furnace arranged for Heating at the Top.**—This gas furnace is exhibited in section by fig. 36: *a* is the gas-burner; *b* is the support for it when used below the furnace; *c* is the iron tripod support for the furnace; *d*, *d*, are two perforated clay plates adapted to the gas-burner *a*; *e*, *e*, are two clay cylinders. These pieces, *a* to *e*, are similar in all the furnaces, and will not require description in each example.

The interior of the furnace, as represented by fig. 36, is built up as follows:—The clay plate, *d*, is put upon the tripod, *c*. Over the central hole in *d* the clay cylinder is placed, and upon that cylinder, two or three of the clay plates. Upon these a porcelain or platinum crucible is placed. If it is of platinum, a piece of platinum foil may be put between the crucible and the uppermost clay plate, to protect the crucible from contact with particles of iron, or against cementation to the clay. The crucible is to be surrounded by the plumbago jacket. The space between this pile in the centre of the furnace and the two cylinders, *e*, *e*, which form the walls of the furnace, is to be filled with flintstones or gravel, washed clean and dried. The stones which answer best are rounded, water-worn pebbles, of half an inch to one inch diameter. These may be piled up to the top edge of the jacket.

FIG. 36.

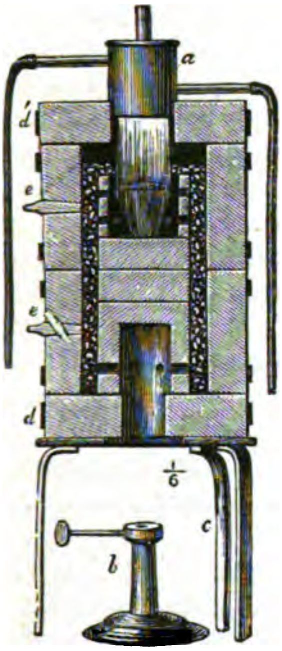


FIG. 37.

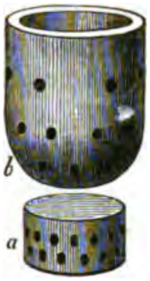


FIG. 38.

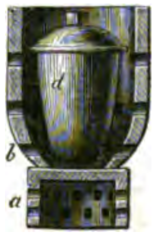


FIG. 39.

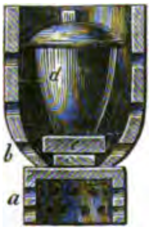
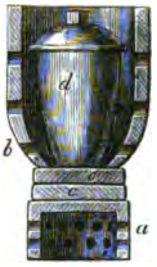


FIG. 40.



It has been found convenient to give the crucible jacket a conical form, the better to adapt it to the usual shape of the crucible. The four figures, 37, 38, 39, 40, show the method of using it so as to make crucibles of different sizes fit the furnace properly.

In these figures *a* represents a ventilator or hollow support, the sides of which are pierced full of holes. This is placed over the hole in the lower nozzle plate, to permit of the descent and escape of the carbonic acid gas and steam produced by the combustion of the gas in the furnace: *b* represents a cone open at both ends and pierced full of holes. Its use is to contain the crucible that is to be exposed to heat, as represented by *d* in figs. 39, 40, 41.

The ventilator and cone together should be equal, or nearly equal, to the height of the body of the furnace: the top of the crucible should be *about* 2½ inches from the flat iron face of the gas-burner, that being in general the place of greatest heat, but this is subject to a variation of ½ inch more or less, according to the supply of gas. The space between the crucible and cone should be about ¼ inch; if much wider the heating power of the furnace is diminished. The space between the ventilator and cone, *a*, *b*, and the sides of the furnace, must be completely filled by flints of from ½ inch to 1 inch diameter. When the flints split up, the powder produced must be occasionally removed, as it stops the draught of the furnace. In order to raise the crucible to the proper distance from the face of the burner, round clay plates are used: thus, *c* (fig. 39) shows how to raise a crucible within a cone; and *c* (fig. 40) shows how a small cone can be raised above the ventilator to the proper height. Different sizes of cones may be used in the same furnace, the cone being chosen in each operation to fit the crucible, the quantity of surrounding pebbles being of no consequence, provided the furnace is filled up to the edge of the cone.

**The Process of Fusion.**—The apparatus being thus arranged, the gas is to be turned on, and lighted; the blowing machine is then to be put into action, and the nozzle of the gas-burner depressed into the central hole of the clay plate *d′*, as shown in fig. 36. The whole force of the blue flame then strikes the crucible; part of it forces its way through the holes in the cone or crucible jacket, and part of it rises and passes over the upper edge of the jacket; after which it finds its way downwards between the pebbles. The carbonic acid gas and the vapour of water which result from the combustion of the gas, together with the nitrogen of the air and any uncombined oxygen, accompany it. No space being left open for the escape of these gases at the upper end of the furnace, they go downwards through the interstices among the pebbles, and passing through the holes in the ventilator *a* (fig. 37), and through the central hole in the lower plate *a* (*fig.* 36), they escape finally into the air. In this progress, the hot gases give up nearly all their heat to the flint stones. Water and gases escape below at a very moderate temperature: water even runs down in the liquid state, while the stones rapidly acquire a white heat, and if the blast and the supply of gas are continued they retain that white heat for any desired length of time—for hours.

**Precautions to be observed on commencing a Fusion.**—When a furnace and its contents are cold, and a burner is newly lighted, it must not be suddenly plunged into the furnace, and the full heat be applied at once; otherwise the fire is apt to go out, or the crucibles and interior fittings to crack from the too sudden application of a violent heat. It is better to let the flame play a little time into the opening of the furnace, before the burner is thrust closely into its place. The crucibles and furnace fittings should be quite dry when used. It is recommended, after arranging a furnace for a fusion, first to warm it by a large gas-burner, before applying the blast-burner. When the furnace has been warmed the full heat may then be applied safely. At the end of ten minutes after lighting the gas, the crucible, placed in the described circumstances and exposed to the full action of the heat of the gas, and surrounded by substances which are bad conductors of heat, is raised, with the jacket and pebbles around it, to a white heat. The consequence is that the full power of the gas jet is then exerted upon the crucible and its contents, and those effects are produced which will be described presently.

If it is desired to inspect the substance subjected to the action of heat in this furnace, the gas-burner is lifted out, and the crucible is examined through the hole in the clay plate. To make it possible to inspect substances at a white heat, the view is taken through a piece of dark cobalt blue glass. If the substances submitted to heat suffer no harm from the action of oxygen, it is better to dispense with a crucible cover and to direct the jet of flame directly down upon the substance to be heated. The action is then more rapid. When the burner is taken out, the substance in the crucible can be stirred, if it is considered necessary.

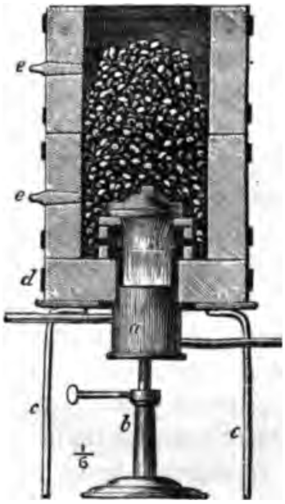
**Results.**—The following experiments will give an idea of the power of a furnace of this description. A common clay crucible, 3 inches high and 3 inches diameter at the mouth, was filled with about 24 ounces of cast iron. It was mounted like fig. 36 in a furnace of 4 inches internal diameter and 8 inches deep. The pebbles were filled in to the edge of the crucible. No crucible cover and no jacket were used. The flame was thrown directly upon the iron. In a short time the iron melted; the oxygen then converted some of the cast iron into magnetic oxide of iron, which formed a thin infusible mass on the surface of the cast iron. At twenty minutes from the lighting of the gas, the furnace was dismounted. The crucible was taken out. A hole was broken by an iron rod in the infusible surface of oxidised iron, and the fused cast iron below it was decanted into a mould, and made a clear casting weighing 20 ounces. In the same small furnace 32 ounces of copper were fused in fifteen minutes. When the furnace is hot, that quantity of copper or cast iron can be fused in ten minutes. In a furnace of the same dimensions, but with a gas-burner having only six instead of sixteen jets, 16 ounces of copper or of cast iron can be completely fused in ten minutes, if the furnace is cold, and in seven minutes if the furnace is hot.

These experiments show that within twenty minutes a heat is producible in this little furnace which is more than sufficient for most assaying or metallurgical operations.

**Gas Furnace heated at the Bottom.**—This is exhibited in section by fig. 41.

In this furnace the parts marked *a*, *b*, *c*, *d*, *e*, *e*, are the same as those similarly marked in fig. 36; but the gas-burner is in this case put into the bottom of the furnace, instead of the top, and the arrangement of the crucible and its support is altered in the manner shown by the figure. Upon the centre of the clay-plate, *d*, the perforated plumbago cylinder is placed, and upon that a plumbago crucible. These are placed together in position in fig. 41. The size of the crucible and the height of the perforated cylinder are to be so adjusted that the bottom of the crucible shall be struck by the hottest part of the gas flame: that is to say, the space left between the face of the gas-burner and the bottom of the crucible must not exceed 2½ inches. The crucible is provided with a closely-fitting cover, and pebbles are then filled in between the crucible jacket and the furnace cylinder *e*, and are covered over the crucible until both the pieces of the furnace, *e e*, are nearly filled. The gas is then lighted, the blast of air is set on, the gas-burner is forced up into the hole in the clay plate *d*, and the operation proceeds. In from ten to twenty minutes after the gas is lighted—this difference of time depending upon the size of the furnace and the weight of metal contained in the crucible—the interior of the lower cylinder *e* acquires a white heat. The progress of the operation can be watched by occasionally removing the stone peg in the trial-hole of the furnace cylinder *e*. The heat very slowly ascends into the upper cylinder, and it never becomes so great in the upper as in the lower cylinder. The greatest fusing power of the furnace is confined within a vertical space of about 6 inches, reckoning from the bottom. The power of flint pebbles to abstract heat from the gases which pass through this apparatus is quite remarkable. When about 6 inches of pebbles lie above the crucible, and the crucible and the

FIG. 41.



pebbles about it have been white-hot for half an hour, the hand can be held over the top of the furnace within a few inches of the pebbles without inconvenience. It becomes wetted with the vapour which rises from the furnace, but feels only a moderate degree of heat.

This form of the furnace is attended by the inconvenience that we cannot examine the condition of the matter contained in the crucible, to ascertain when the heat has been continued long enough. In cases where the fusion is performed repeatedly on the same weight of metal, this would be of no importance, because the power of the furnace is so steady and regular that the time of firing which has been found to answer once will answer the same purpose again.

When it is supposed that the fusion of the metal submitted to trial is completed, the gas is first to be turned off, and then the supply of air stopped. We can either allow the furnace to remain intact till it is cold, or lift off the cylinders *e e* with tongs, and allow the hot stones to fall into the iron pan placed below the furnace to receive them. A few bricks should be laid between the pan and the table or stool on which it rests, if the latter is made of wood, because the heat given off by the pebbles is very great. The pebbles being raked away from the crucible, the contents of the latter can be examined.

The absolute sizes of the furnaces depend upon the amount of work required from them. The fusions described below were mostly made in a furnace of 6 inches internal diameter, a few in a furnace of 4 inches internal diameter and one or two in a furnace of 8 inches internal diameter; all of them with a gas-burner of 16 holes and a supply of gas obtained from a ½-inch pipe. A large furnace with an internal diameter of 12 inches, will demand a gas-burner of 26 holes, and a supply of gas from a pipe of nearly 1 inch in the bore.

**Examples of Fusion effected by the Blast Gas Furnace.**— The fusing points of certain metals have been fixed by Daniell at the following temperatures:—

| | °F. | | °F. |
|---|---|---|---|
| Silver . . . | 1873 | Copper . . . | 1996 |
| Gold . . . | 2016 | Cast-iron . . . | 2786 |

**Brass, with 25 per cent. of zinc, at 1750° F.**

All these metals melt readily in the gas furnace. Quantities of 3lbs. of copper or cast iron can be completely fused in fifteen minutes in a 6-inch furnace. Quantities of 8 lbs. or 10 lbs. of copper or cast iron can be completely fused into a homogeneous mass in a 6-inch or 8-inch furnace within one hour, using a 16-hole burner, and a supply of gas from a ½-inch pipe.

In a furnace of the same size 45 ounces of nickel have been fused, and in other experiments masses of wrought iron, weighing 18 ounces, 28 ounces, and 40 ounces, have been produced. The piece of 18 ounces was perfectly fused. The piece of 40 ounces was not quite fused, the crucible having melted and stopped the operation. Cobalt has also been fused, and reduced to the metallic state from the peroxide by ignition with charcoal. The time required for the fusion of these refractory metals is from one and a half to two hours.

Scraps of platinum can be fused into a porous mass, but not into a solid homogeneous bead. Thin platinum wires fuse readily in the free flame of the gas-jet produced by the burner fig. 34; but when the jet plays upon a quantity of the metal contained in a crucible, the relations of power and effect are different.

When the metals to be melted are such as do not undergo oxidation, the method of action represented by fig. 36 is most convenient. In this manner gold can be readily melted, and by removing the gas-burner the melted metal can be stirred. When the action of oxygen is to be avoided, the crucible must have a cover, which in some cases should be securely luted to it.

**Choice of Crucibles.**—The experiments above referred to were made with coal gas at the ordinary pressure, and with a blast of cold atmospheric air. Greater effects can be produced by the use of oxygen gas, or of heated atmospheric air. But a difficulty stands in the way of the use of these greater degrees of heat in the want of crucibles capable of enduring their action.

With cold atmospheric air, pure nickel and pure iron attack every kind of siliceous crucible, and it is therefore needless to heat the air or to prepare oxygen till a superior kind of crucible is obtainable. At present, these metals can only be melted in plumbago crucibles, which necessarily communicate to them more or less carbon. Metals which melt at moderate degrees of heat, such as gold and copper, are easily fused either in clay crucibles or in those of plumbago—the latter, be it remembered, being a mixture of graphite and clay. Metals in combination, such as cast iron, also melt readily in clay crucibles without destroying them. But when such metals as iron, nickel, and cobalt, are freed from carbon, and brought into a state of purity, they acquire an extraordinary attraction for silica at a white heat, so that the metal and the silica readily run down into a very fusible silicate. Even when plumbago crucibles are used, the carbon burns away at some particular point; the metal then attacks the clay, bores a hole through the crucible, and finishes the operation.

No kind of clay or porcelain will withstand the action of pure iron or nickel at a white heat; it is therefore almost impossible to effect any large fusions of these metals when they are free from carbon.

**Fusion of Metals in large quantities, and Ignition of Objects of large size.**—As the gas-burner fig. 34 can be held in any required position, it is possible to apply heat to large objects by using several gas-burners. Thus a large crucible may be fixed in a square furnace, and gas-burners be applied below and on the four sides of the furnace, the spaces between the crucible and the walls of the furnace being filled with pebbles, to collect the heat and apply it to all parts of the crucible.

**Muffle Furnace for Assaying, Roasting, etc.**—A muffle placed in an assay furnace, and built up with pebbles, can be heated either from above or from below by the blast gas-burner. The flame and products of combustion can be made to sweep through the muffle, whether going upwards or downwards. The *air*-pipe and *gas*-pipe attached to the gas-burner (fig. 34) must each be provided with a stop-cock. When the front door of the muffle is opened to afford the opportunity for examining the cupels, the blast, if continued, would blow these out against the operator, but that occurrence is prevented by turning the stop-cocks. When it is desired to oxidise the substances in the muffle, the furnace is first brought up to a sufficient temperature, and then the gas is turned off, but the blast of air is continued. The air passing through the hot pebbles enters the muffle at a high temperature, and not exhausted of oxygen, because there is no carbonaceous matter present among the pebbles when the gas is turned off. The pure and highly-heated air is consequently in a proper position for oxidising metals that are already raised to a red heat in the muffle. The same apparatus is useful where substances require to be roasted in the presence of air in order to oxidise and expel some volatile ingredient. We have in this process an effectual means of using hot air to aid the process of cupellation.

**Miscellaneous uses of the Blast Gas Furnace.**—1. The preparation of chemical substances by the projection of mixtures into a crucible kept at a red or a white heat. 2. For melting silver, gold, copper, cast iron, brass, bronze, nickel-silver, &c., either for making small castings or ingots. 3. For experiments on glass, every description of which it is able to fuse. 4. For experiments on enamels, coloured glasses, and artificial gems. 5. For experiments on metallic alloys. 6. For the fusion of steel. 7. For the use of dentists, in the preparation of mineral artificial teeth. 8. For the assay of ores of silver, copper, lead, tin, iron, and other metals. 9. For all purposes of ignition, combustion, fusion, or dry distillation, at a red heat or a white heat, where it is desirable to produce those temperatures promptly and cheaply.

**Repair of the Gas Furnace.**—When the clay cylinders become warped or chipped, so as to allow the gases to escape at the joints laterally, they must be luted for each operation by applying a little wet fire-clay by means of a spatula. When only a moderate heat is required, this luting is unnecessary.

Many chemical ignitions performed on small quantities of substances in analytical processes, demand a degree of heat greater than that afforded by gas-burners with draught, but not so much as is supplied by the powerful blast-furnaces described in the preceding section. Mr. Griffin has therefore described an apparatus intended to meet this requirement. It will readily raise to a white heat all the sizes of platinum and porcelain crucibles that are commonly in use, and such sizes of clay or plumbago crucibles as will contain a pound of cast iron, which quantity of metal this furnace will melt. Figs. 42, 43, 44, represent three examples of the miniature blast furnace. This consists, in the main, of a blast gas-burner, similar in construction to that represented by fig. 34, but smaller in size, and having only three jets. It is fixed upon, and forms part of, the furnace support, as represented in the figures. Upon the iron nozzle of the burner there is fixed a fire-clay nozzle-plate or furnace-sole, similar to *d* (fig. 36), and upon this plate the little furnace is built up of loose clay cylinders, which are in all cases selected to suit the size of the crucible that is to be operated upon. The entire furnace rests on the solid shoulder of the gas-burner. No pebbles are used, the degree of heat that is intended to be raised not requiring their aid. Gas supplied at common pressure by a ¼-inch pipe is sufficient.

In mounting this furnace, it is necessary to place between the nozzle-plate or sole, and the conical flue placed upon it, three small feet, to separate the two pieces, and give room for the influx of atmospheric air around the flame, without which the proper heat of the furnace is not obtained. Three bronze halfpenny pieces answer the purpose exactly.

Upon comparing the three figures it will be perceived that the interior of the furnace is exactly alike up to the grate or trivet, and differs above that only in having cylinders that suit the different sizes of crucibles that are to be heated.

It is in the power of the operator, when working with platinum crucibles, to dispense with the grate and to hang his crucible in the hottest part of the furnace by a sling of platinum wire suspended from an iron bar laid across the top of the furnace. The piece marked *u* (in fig. 42) being omitted, the two pieces *e e* come together and form a cavity, in the centre of which the crucible is to be suspended. If it then appears to be too low in the flame, the height can be raised by putting such pieces as *a* or *c* (fig. 54) between the lower piece *e* and the sole. For the above description, and many of the cuts, we are indebted to the kindness of Mr. Griffin.

G. Gore, Esq., F.R.S., has devised a gas furnace which

Fig. 42.

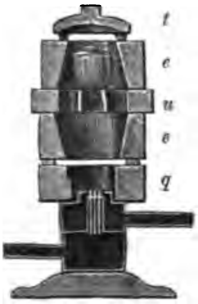


Fig. 43.

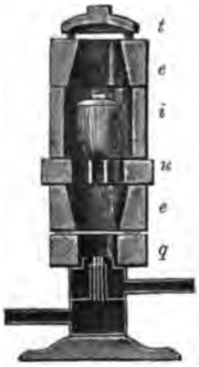
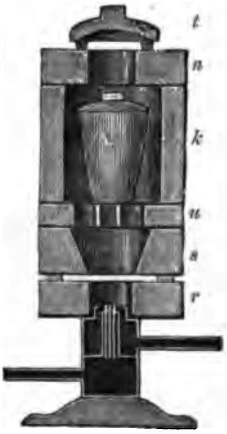


Fig. 44.



will fuse cast iron, &c., and still allow the melted substances to be perfectly accessible to be manipulated upon for a continuous and lengthened period of time, without contact with impurities or with the atmosphere, and without lowering their temperature sufficient to cause them to solidify. These conditions Mr. Gore has obtained by means of ordinary coal-gas and atmospheric air, without the use of a bellows or lofty chimney, or of regenerators or valves requiring frequent attention. The arrangement is as follows: *A* (figs. 45 and 46) is a cylinder of fire-clay about nine inches high and six inches diameter, open at both ends, with a hole in its side near the bottom to lead into the chimney; it is covered by a movable plate of fire-clay, *B*, with a hole in its centre for introduction or removal of the crucible, &c.; this hole is closed by a perforated plug of clay *C*, for access to the contents of the crucible, and that again is closed by another clay stopper *D*: *E* is a chimney of sheet iron about five or six feet high, kept upright by a ring of iron *F* attached to the top of the furnace. The fire-clay cylinder is closed in a sheet iron casing with a bottom of iron, to which are fixed three iron legs *G*. An iron tube *H*, with a

FIG. 45.

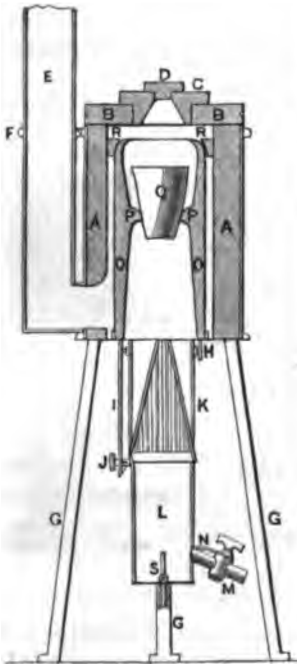
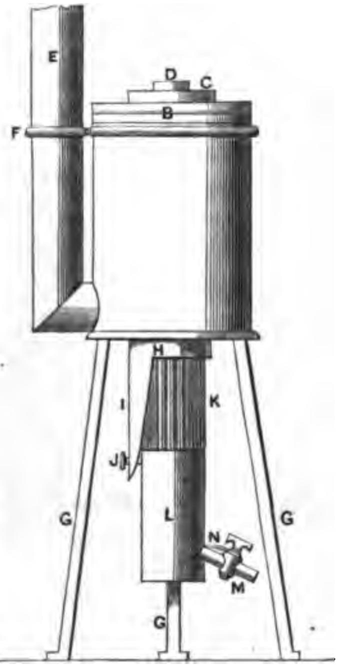


FIG. 46.



prolongation *I*, supports by means of the screw *J* the burner *K* and its tube *L*, which is open at both ends. Gas is supplied to the burner by means of the tap *M*, which has a small index *N* attached to it for assistance in adjusting the gas. Inside the large cylinder is another fire-clay cylinder or cupola *O*, with open ends, and with three projections of fire-clay *P* for supporting the crucible *Q*; it is kept steady by means of three clay wedges *R*; *S* is an air-valve for closing the bottom of the tube *L*. The gas-burner is a thin metal cylinder, deeply corrugated at its upper end, with the corrugations diminishing to nothing at its lower end, as shown in the engravings. The action of this furnace is as follows: Gas is admitted to the open tube *L* by the tap *M*; it there mixes with air to form a nearly combustible mixture, which ascends through the burner, and burns in the clay cylinder *O*, being supplied with the remainder of air necessary to combustion through the tube *H* to the outer surface of the flame; the products of combustion pass up through cylinder *O*, and then downwards outside of it to the chimney, the point of greatest heat being at *Q*.

It is important in using this furnace that the burner is placed quite in the centre of the bottom of the tube *O*; also that a crucible of not too large nor too small dimensions be selected. The most suitable way of supporting a smaller crucible is by placing it in a larger one that has had its upper part broken off. If desirable, a little clay luting may be placed round the top edge of the iron casing to exclude air entering between it and the cylinder; also a little thin clay luting upon the part of the bottom of the furnace where the inner cylinder *O* rests.

In lighting the furnace, the plugs *C* and *D* are removed, a light held *inside* the opening, and the gas turned on full. Should the flame blow down to the bottom of the tube *L* on lighting (which, however, rarely occurs unless the furnace is already hot), the gas must be turned off, and the bottom of *L* momentarily closed whilst lighting the gas as before. Should the flame not burn down to the burner but only burn to the orifice in the clay plate *B*, it must at once be extinguished and relighted, otherwise some of the gaseous mixture will pass into the chimney unburned, and subsequently ingnite and cause an explosion. A large flame now issues from the top orifice, and is white if too much gas is on, and chiefly violet or red with the proper quantity; it should now be coarsely adjusted until these appearances are represented. The annular plug *C* should now be inserted, which will compel it to pass downwards to the chimney, and as soon as the small remaining flame now issuing disappears, or nearly disappears, as it will in a few seconds, the small stopper *D* should also be inserted. In lieu of this, the large flame may be deflected against the chimney by means of a piece of sheet iron until it withdraws inwards as before mentioned; the two plugs may then be reinserted. The gas tap may now be partly adjusted. The crucible should be placed in the furnace after the act of lighting the gas, but not immediately after if the furnace is cold, or explosions may occur by unburned gaseous mixture passing the crucible into the chimney, and igniting afterwards.

After about five minutes the gas should be slowly adjusted, until a sound is heard inside like a series of small explosions. This sound is sometimes not very distinct, especially at high temperatures, and therefore requires a little experience in the use of the furnace in order to be detected. It is, however, a chief guide in determining the proper amount of gas, and should therefore be carefully studied. To assist in adjusting the gas it will be found very useful to place a small piece of looking-glass beneath the tube *L*, and to adjust the gas tap until the flame between the burner and crucible appears wholly violet or slightly white; but this test is liable to fallacy if employed when the gas is first lighted, because the coldness of the parts makes the flame much whiter than it otherwise would appear. It is also fallacious, the flame appearing whiter than it really is when the crucible is very hot. It is, however, of great assistance, especially at intermediate temperatures. A rough deposit upon the outer edge of the crucible indicates an excess of gas; the deposit is carbon. Less gas is required with a crucible in the furnace than without one; also less is required when the small hole at the top of the furnace is open than when it is closed; and less is also required when the furnace is cold than after it has been lighted some time, because the draught gradually increases and draws in more air. After having accurately adjusted the gas, no further attention to the furnace is requisite.

Having once found the proper adjustment of gas under certain known conditions, it is well to notice the position of the index pointer *N*, in order to be able at once to adjust it to about the right point on other occasions. Under ordinary circumstances, during daylight it is best to set the gas nearly full on at first, and fully on at about five minutes afterwards when the draft has become more powerful; but during twilight, when the supply of gas from the gas works is more free, the index pointer may be set at the numbers 2½ or 3. The gas should be supplied by a pipe of not less than ⅜-inch bore, with a main pipe of ½ an inch; but all depends upon the pressure of gas at the particular locality, which is very variable. The consumption of gas varies from 30 to 40 cubic feet per hour, the value of which is about twopence.

The top of the chimney should be placed in a position where the products of combustion can pass freely away. If it is placed in an opening or pipe leading to another chimney, care must be taken not to have the draught too powerful, otherwise the heat will be drawn more into the chimney, and the supply of gas in the daytime may be found rather deficient. The furnace will act satisfactorily, though less powerfully, with the chimney standing in an open room without any special outlet for the products of combustion, provided the full height (6 feet) of chimney is employed. Under other circumstances a chimney 4½ or 5 feet high may be used.

This furnace will produce what is generally called a white heat; it will readily melt half a pound of copper, or six ounces of cast iron; it will melt as large a quantity of those substances as the largest sized crucible that can be introduced into it will contain, sufficient space being reserved around the crucible for draught. It requires from 20 to 30 minutes to acquire its highest temperature, and then the entrance part of the chimney exhibits a faint red heat in daylight. If it exhibits much more than this the draught is too powerful, and if less, there is not sufficient gas.

With one ounce of copper put into the cold furnace, and the gas lighted and properly adjusted, the copper generally begins to melt at about the tenth or twelfth minute, and is completely melted by the fifteenth. With the heat well up, 1 ounce of copper has been melted in it in 2¼ minutes, 1 ounce of cast iron in 3 minutes, 5 ounces of copper in 4¼ minutes, and 3 ounces of cast iron in 5 minutes.

With the smaller hole in the top of the furnace open, 1 ounce of copper has been melted in 3½ minutes, and several ounces of copper have been kept in fusion upwards of half an hour, and may be kept so for any length of time; cast iron has also been fused, and kept melted under the same conditions. These various effects have also been obtained in a somewhat diminished degree with the chimney standing in an open room.

When the small hole *D* is open some air is drawn in that way, and less air passes up with the gas through the tube *O*, but the cold air does not much diminish the temperature of the crucible, because it combines with the excess of gas now passing over the edge of the inner cylinder; it, however, renders the flame round the crucible white by deficiency of air, and this should be partly corrected by lessening the gas. An excess of either gas or air renders the surface of melted copper dull.

When it is desirable to perfectly avoid contact of air with the fused substance during manipulation, a narrow crucible should be employed, and a thin narrow ring of fire-clay should be placed upon the top of the tube *O* to contract its opening; the flame then closes completely over the top of the crucible and prevents access of air; a proper adjustment of gas, together with exclusion of air in this manner, enables a perfectly bright surface of melted copper, or even tin, to be continuously maintained, from which the images of parts above are clearly reflected. The clay ring may be withdrawn by lifting the plate *B*. A less perfect exclusion of air may be obtained by employing a narrow crucible placed rather low down in its support. A small iron dish should be placed beneath the tube *L*, to receive any melted substance that may fall. The chief conditions of success in the use of this furnace are sufficient gas, a suitable degree of draught, and proper regulation of gas to air.

The advantage which Mr. Gore's gas furnace possesses over those previously described is that no artificial blast is required in using it. Mr. Griffin has since devised what he calls a *Reverberatory Gas Furnace*, which also produces a high temperature without the use of a blowing machine. It is especially suitable for assay purposes on a small scale and for the decomposition of siliceous minerals by fusion with alkaline carbonates in platinum crucibles, being capable of fusing 1,000 grains of anhydrous carbonate of soda in ten minutes.

The different parts of this furnace are also arranged in a very convenient manner, so as to admit of its being employed for purposes in a chemical or assay laboratory. It is based upon a new form of gas-burner which, aided by suitable bellows, can be used as a convenient source of heat for most operations of the chemical laboratory and lecture table. It will boil a quantity of liquid, exceeding two gallons, at once; it will raise a 4¾-inch fire-clay crucible to full redness; it will fuse anhydrous carbonate of soda in greater quantity than is required for the analysis of a siliceous mineral; and it will melt small quantities of sterling silver. This amount of power is sufficient for most chemical and many metallurgical operations.

Fig. 47 represents the gas-burner of this apparatus. The gas is supplied by the horizontal tube, whence it passes through a set of small holes into the box *a*, in which it mixes with atmospheric air that enters freely by the holes shown in the sketch. The gaseous mixture passes up the vertical tube *b*, and is inflamed at the top, where it burns with a single tall blue flame, which gives no smoke, very little light, but much heat. In this condition the apparatus differs from 'Bunsen's gas-burner' only in size. *c* represents a thin brass cap, which fits the air box *a*, but moves easily round it; *d* is a flat cast-iron box with many holes round the margin, and a few small ones on the top. This box fits loosely on the upper part of the tube *b*, and when it is placed upon it and the gas is lighted the flame produced consists of a series of radiating jets, forming a horizontal circular flame of about seven inches in diameter. Fig. 48 *a* shows a ring of horizontal flames produced; 47 *b* gives the single vertical flame.* The ring of flame is suited to the purposes of boiling and evaporation; the single flame to ignition and fusion. The height of the apparatus represented by fig. 47 is twelve inches; the bore of the tube *b* is one inch; and the diameter of the fire-box *d* is four inches.

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* Fig. 48 represents a small variety of this gas-burner, in which the head is not removable, but the efflux of the mixed gases is regulated by a sliding valve, which is represented by *b*.

When a large crucible is to be heated to redness, the gas-burner is to be used without the rose, and is to be arranged with the furnace fittings that are represented in perspective by fig. 49, and in section by fig. 50, and the lower part of fig. 51, *a*, *b*, *c*, *d*. Letter *a* represents the gas-burner; fig. 49 *b* is a tall iron stool; *c* a chimney which collects atmospheric air to feed the flame, and lead it up close to the vertical tube of *a*, by which contrivance the air is warmed and the tube cooled; *d* is a furnace-sole or plate of fire-clay; *f* is a reverberatory dome, the interior of which is best shown in the section fig. 50; *e* is a cast-iron ring or trivet, represented more clearly in fig. 52; *g* is an iron chimney, 24 inches long and 3¼ inches wide; and *h* a damper to lessen the draught when small crucibles are to be heated. The height of this apparatus from *a* to the top of *f* is 24 inches; and the external diameter of the dome *f* is about 8 inches. The crucible, which may be from 4¼ to 4¾ inches in height, is placed on the iron ring *e* fig. 50 or fig. 52, and that on the clay sole *d*, and it is then covered by the dome *f*. The gas should be lighted after the crucible is placed in its position and before the dome is put on. The dome and the chimney are then to be added and the operation allowed to proceed. With a crucible of the above size, the damper *h* is not required; but it must be used when the crucible is under 4 inches in height, otherwise the draught occasioned by extra space within the dome causes the flame to blow down. The damper must be put on the chimney before the chimney is put on the dome. The iron ring (fig. 52 or *e* fig. 50) suits crucibles of different sizes, according to which side of it is turned uppermost.

FIG. 47.

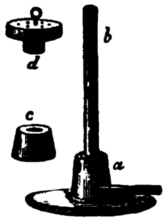
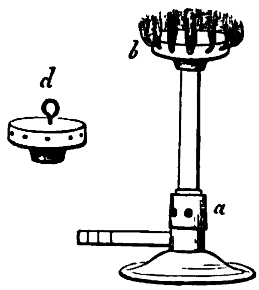


FIG. 48.



The figures show that a crucible mounted in this furnace can lose very little heat by radiation or conduction, and hence it is that a small gas flame produces a powerful effect. In half an hour a 4¾-inch clay crucible, filled and covered, can be heated to full redness. The progress of the ignition can be easily examined by lifting up the chimney *g* and the dome *f* by their respective wooden handles. But the action of the furnace can also be judged of by a peculiar roaring

Fig. 49.

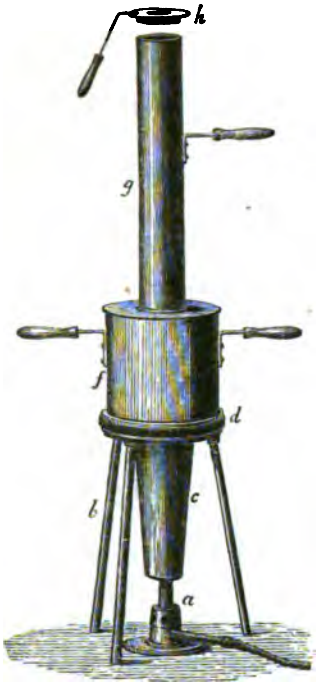


Fig. 51.

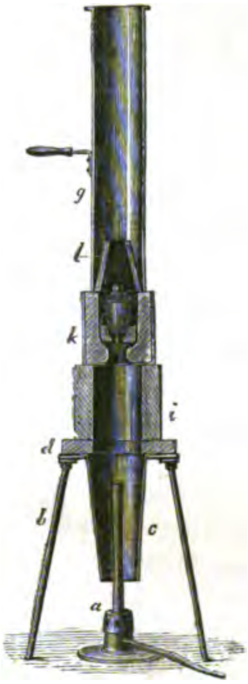


Fig. 50.

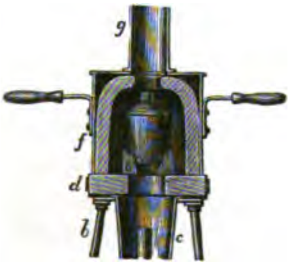
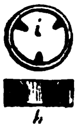


Fig. 52.

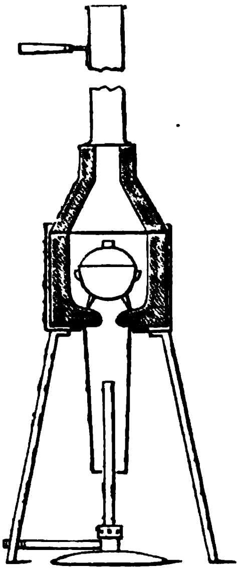


noise which it produces. If the gas and air are mixed in due proportions, the roar is regular and continuous; if there is too much gas the roar is lessened, if too much air the roar is increased, but is rendered irregular and intermittent. The greater the noise, the greater the heat in the furnace. And when the roar becomes spasmodic the flame is on the point of blowing down. To prevent that occurrence, the proportion of air must be lessened or that of gas increased.

The following arrangement is convenient when small crucibles are to be strongly heated: anhydrous carbonate of soda in quantities exceeding 1,000 grains can be thus readily fused in a platinum crucible, and sterling silver can be melted in a clay crucible. It is also available for ignitions or fusions in small porcelain crucibles. Fig. 51 represents the arrangement of apparatus, as seen in section: *a* is the gas-burner; *b* the stool; *c* the air chimney, and *d* the furnace-sole, as already explained; *i* is a cylinder of fire-clay, 4 inches high, and 4½ inches diameter; *k* is a fire-clay furnace, in which is placed a small cast-iron ring about 2 inches in diameter, similar in form to that represented by fig. 52, and on this ring the platinum crucible is adjusted; *l* is a fireclay or plumbago reverberatory dome; and *g* is the chimney that forms part of the furnace represented by fig 49. The crucible being adjusted, the gas lighted, and the dome and chimney put on, the lapse of twelve or fifteen minutes, according to the quality and pressure of the gas, suffices for the fusion of 1,000 grains of carbonate of soda in a platinum crucible. At the heat which this furnace produces the cast-iron ring does not melt nor alloy with the platinum crucible placed upon it.

By a modification of these arrangements, Mr. Griffin has made a gas furnace for melting quantities of lead, zinc, antimony, &c. This is represented by fig. 53. The iron crucible will contain nearly 30 lbs. of lead and about 24 lbs. of zinc. The burner readily melts these quantities, and then, with a diminished quantity of gas, will keep the metals fluid. The metals being protected from the air suffer little loss by oxidation. Such operations as the granulation of zinc are performed with this apparatus with great facility; it serves also for baths of fused metal. In a large furnace of this kind, made for a special operation, 60 lbs. of zinc have been melted with ease, and the inventor believes that, used in this manner, the burner is powerful enough to melt a hundredweight of zinc.

Fig. 53.



The principles of heating by gas, which have led Mr. Griffin to the construction of these gas furnaces, may be summed up as follows: When a crucible or other solid body is to be heated, it is to be wrapped in a single flame at the point of maximum heat, and loss of heat by radiation and conduction is to be prevented by the interposition of non-conducting materials (plumbago or fire-clay); and when liquids are to be boiled or evaporated, particularly when they are contained in vessels of glass or porcelain, the flame is to be broken up into numerous horizontal jets, and these are to be made to supply a large and regular current of highly-heated air, by which alone, and not by the direct application of the flame, the vessel that contains the liquid is to be heated. In both cases provision must be made to secure a sufficient draught of air through the furnace, because every cubic foot of gas requires for combustion 10 or 12 cubic feet of air, and the gases which have done their duty must be rapidly carried away from the focus of heat. If the steam, the carbonic acid gas, and the free nitrogen which constitute the used-up gases are not promptly expelled, fresh gaseous mixture in the act of producing additional heat by combustion cannot get near the object that is to be heated, and the heat so produced out of place is wasted.

Bunsen's gas-burner, whatever its size, is subject to two defects: sometimes the flame burns white and smoky, and sometimes it blows down, the gaseous mixture explodes, and the gas then burns with a smoky flame in the tube. The remedies for these defects are as follows: If the flame is white only when the gas is turned on very full, the remedy is to lessen the supply of gas; but if the flame continues to burn white at the top when the gas is gradually turned off and the mass of flame slowly sinks, then the holes which deliver the gas from the supply pipe into the air-box *a* (fig. 47) are too large, and are placed too directly under the centre of the vertical tube *b* (fig 47), and these defects must be corrected in the instrument. Finally, when the flame blows down it is because the supply of atmospheric air is too large in proportion to the supply of gas, and their relative proportions must be altered. To effect this alteration the cap *c* is to be turned round on the air-box *a* so as partially to close the holes, and thus lessen the supply of air. If, when the gas is alight the flame needs to be lowered, first the supply of air is to be lessened and then the supply of gas. If the flame is to be enlarged, first the supply of gas must be increased and then the supply of air. In short, to prevent the flame blowing down, the gas must always be placed in excess, and then have the proper quantity of air adjusted to suit it by means of the regulator *c*. When gas-burners of this description have to be used in a locality where the pressure of the gas is slight, especially in the daytime, there is a constant tendency in the flame to blow down. The best way to prevent that occurrence is to supply the gas by a pretty wide tube, and to see that the current of gas is not checked by a very narrow bore in the plug of an intervening stop-cock, which is often the unsuspected cause of want of pressure in the supply of gas. If this does not suffice to prevent the blowing down of the gas, the holes which admit the gas from the supply pipe into the box *a* of the burner should be enlarged, more or less according to necessity. A large supply of gas compensates, to some extent, for want of pressure.

When a steady and long-continued heat is desired from a Bunsen's burner it is proper to use two stop-cocks and a length of caoutchouc tube between them. One of these stop-cocks is to be affixed to the burner, and the other to the supply pipe. The latter is to be opened *wider* than is necessary to supply the required quantity of gas, and the former is to be used to regulate the supply to the burner exactly; under these circumstances, if another stop-cock is opened and gas burnt in the immediate neighbourhood, the flame does not so readily blow down in the regulated burner as it does when only the stop-cock on the supply pipe is used.

When a crucible is suspended by wires or by a ring over the flame of a spirit lamp or gas-burner, the flame and the hot air supplied by the flame strike the crucible for an instant and then pass away to do no more good. At the same time the effect of the heating power on the crucible is lessened by other circumstances; namely, by radiation on all sides, by a mass of cold air which constantly rises around and in contact with it, and by the conducting power of the metallic apparatus which supports both the crucible and the lamp. These losses are avoided if the crucible is enclosed in a furnace made of a non-conducting material, such as fire-clay, which can absorb and retain heat. In the descriptions of the gas furnaces, and in that of Charles Griffin's oil-lamp furnace, several methods of mounting crucibles in fire-clay jackets have been shown; and we will now describe some of Mr. Griffin's fittings that may be used to construct temporary table furnaces for crucibles that are to be exposed to the flame produced by gas, oils, or spirit, up to a temperature close upon, but not quite up to, a white heat; that is to say, up to a heat that will readily melt anhydrous carbonate of soda and small quantities of silver, and so be fit for many metallurgical operations, but which will not melt copper nor cast-iron.

Fig. 54 represents sections of cylinders of fire-clay which are drawn on a scale of 1 to 8, and have the relative heights and bores represented in the figures. The clay pieces, that is to say, as many of them as are necessary for a given purpose, can be adjusted over a gas-flame by means of a tripod (fig. 49) or a clay support.

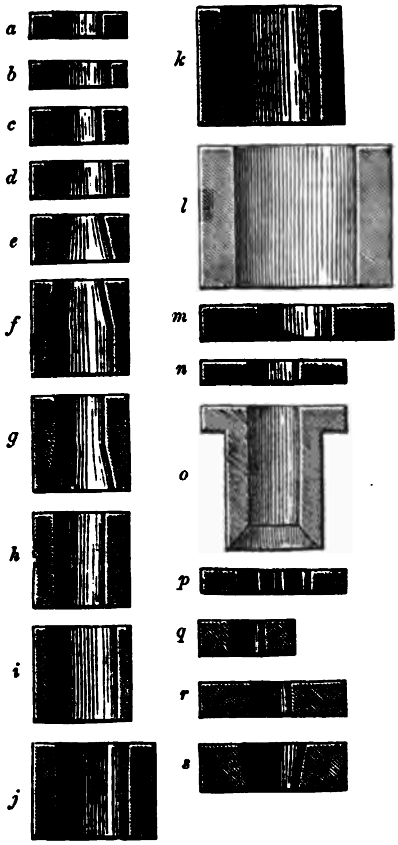
The crucible to be operated upon is to be supported on a toothed ring made either of cast iron or fire-clay, such as are represented by figs. 52 and 55. Fig. 52 is a ring of cast iron, *h* representing it in section and *i* as seen from above. It is about two inches in diameter, and has three teeth projecting towards the middle of the ring. This ring can be supported by any of the clay cylinders whose bore does not exceed two inches. Fig. 55 is a ring of fire-clay of 4 inches external diameter, and 1 inch in thickness, provided with three teeth that project inwards and upon which a crucible can be supported without injuring the draft of the gas furnace.

Both these grates will support crucibles at the highest temperature which can be produced by spirit, oil, or gas, without a blast of air; but at a white heat produced by any of these fuels with a blast of air, the iron ring melts, and if the heat is long continued, those of fire-clay soften and partially give way. When the fire-clay grate (fig. 55) is required to sustain a very high temperature for a considerable time, it is proper to have it made of 6 inches diameter, as represented by fig. 54 *p*, the air-way in which is the same as that of the small grate, but the clay ring is much stronger.

The grate is fixed above the flame at a distance which is found by trial to place the crucible on the point of greatest heat. Commonly a 4-inch cylinder (54 *h* or 54 *g*) placed upon a suitable support serves the purpose. The bore of the cylinders at the bottom must be wider than the burner, to allow of a considerable influx of atmospheric air around the flame. The grate is placed on this cylinder, the crucible on the grate, and then another cylinder around the crucible. The choice of this upper cylinder depends entirely upon the size of the crucible that is to be heated. Whatever the size of the crucible, the cylinder must be so chosen as to fit the crucible as accurately as possible, leaving between it and the furnace walls an open space of not less than $\frac{1}{8}$ inch, nor more than $\frac{1}{5}$ inch all round. If the upper cylinder is not contracted at top like 54 *e f g*, then a cylinder of narrow bore, such as 54 *a* or *c*, must be put upon it, in order to deflect the flame, and the rising current of hot air upon the top of the crucible, and thus produce a reverberatory furnace. Finally, an iron chimney, 2 or 3 feet long, must be put upon the furnace, to force up a draft of air sufficient to feed the flame.

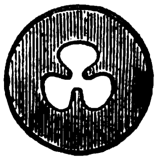
Suppose a small rose gas-burner is to be arranged for an ignition, with the use of a fire-clay support, the combination of pieces necessary for the purpose may be those represented by fig. 56, where A is the fire-clay support, and the rest of the pieces are those which are shown at fig. 54, and described at the letters placed against each of them in this figure. It is evident that the application of this furnace to crucibles of different sizes depends upon the proper choice of the cylinders here marked *i* and *e*. Of course there is only a limited choice of crucibles suitable for such operations. Three inches is the extreme width between the furnace walls of any of the pieces in fig. 54, from *a* to *g*, and though larger cylinders could be used, such as *i* to *o*, it must be remembered that the flame of a lamp without blast has only a limited power, and that although a given flame will fuse 1,000 grains of carbonate of soda in a platinum crucible, it may only heat to a moderate redness a large clay crucible. Yet, considering that low degrees of heat are suitable for many purposes, it is convenient to have the power of readily adjusting a temporary furnace to the bulk of any crucible which it is desired to heat.

FIG. 54.



The clay pieces (fig. 54 *i* to *p*) are thos that have been expressly designed for the blast oil furnace already described; but these can also be used for spirit and gas furnaces, the respective sizes being chosen in each case according to the size of the crucible that is to be ignited.

FIG. 55.



In respect to the *means of supporting* a crucible, it has been shown that clay trivets with a wide flange, namely, the 6-inch trivets fig. 54 *p*, will support a crucible containing 5 lbs. of iron until that quantity of iron is melted, even under the operations of a blast: so that it is evident that this method of supporting a crucible in a gas flame may be always depended upon when no blowing-machine is employed. The discovery of the fact that a trivet of fire-clay of the form of fig. 55 could sustain a crucible bearing 5 lbs. of cast iron, until that quantity of iron has melted under the action of a blast, induced Mr. Griffin to make some experiments on the joint use of a small blast gas-burner and the small fire-clay cylinders that are here described, and these experiments led to the construction of the *Miniature Blast Gas Furnace* already described, an apparatus that justifies the recommendation that has been given of the use of these cylinders; for in the miniature blast gas furnace, the chemist has an instrument which possesses great power in a small compass, and convenient form, the cost of which is a trifle, and which by the addition or exchange of a few fire-clay cylinders can be modified to suit a great variety of operations at high temperatures.

FIG. 56.

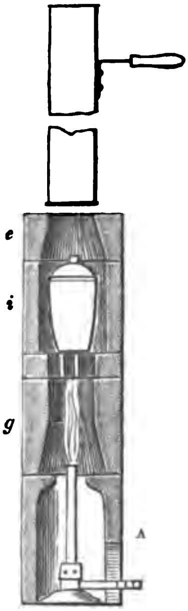


FIG. 57.

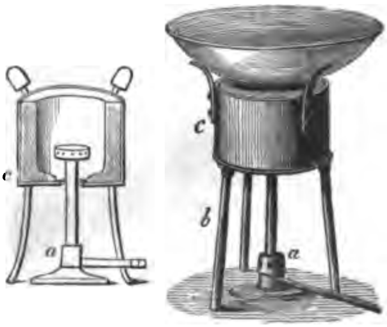


Fig. 57 represents the gas-furnace arranged for boiling the evaporation: *a* is the gas-burner; *b* an iron stool with three legs; *c* a furnace body or iron jacket lined with plumbago or fireclay. This furnace may be 14 inches high and 9 inches in diameter. The three brackets fixed on the upper part of the jacket serve to support the vessel that contains the liquid that is to be boiled or evaporated. A porcelain basin of 16 or 18 inches in diameter can be thus supported. It is important to allow between the jacket *c* and the evaporating basin plenty of space for the escape of the heated air, which ascends from the interior of the furnace. When the evaporating basin is of small diameter, it may be supported on iron triangles, placed in the furnace *c*. The section shows that around the vertical tube of the gas-burner *a* there is in the bottom of the furnace *c* a circular opening which is of 2 inches diameter, and through which air passes freely, partly to feed the flame and partly to be heated by the flame and be directed upwards in a continuous current upon the lower surface of the basin that is to be heated. The flame within the furnace burns steadily. No side currents of air agitate it. No part of it touches the basin, which should receive its heat solely from the mass of ascending hot air. The gas-burner thus arranged and supplied by a gas pipe of ¼-inch bore, burns about 3 cubic feet of gas in an hour, and the flame which it produces, acting upon water contained in an open porcelain evaporating basin, will heat from 60° to 212° F.—

| | |
|---|---|
| 1 quart in | 5 minutes |
| 1 gallon in | 15 ” |
| 2 gallons in | 30 ” |

and when the water boils it is driven off in steam at the rate of more than a gallon of water per hour. The method is consequently applicable to distillation on a small scale, and to numerous other laboratory operations.

**Lutes and Cements.**—It may be as well to mention in this part of the work the various lutes and cements which may be employed, either in fire operations or in making good joints in experiments with gases or liquids. The following are the principal kinds: The best fire lute is that described by Mr. Parker, and is composed of good clay two parts, sharp washed sand eight parts, horse-dung one part. These materials are to be intimately mixed; and afterwards the whole is to be thoroughly tempered like mortar. Mr. Watt's fire lute is an excellent one, but is more expensive. It is made of finely-powdered Cornish (porcelain) clay mixed to the consistence of thick paint with a solution of borax in the proportion of 2 ounces of borax to a pint of hot water.

**Fat Lute** is prepared by mixing fine clay, in a fine powder, with drying oil, so that the mixture may form a ductile paste. When this paste is used the part to which it is applied ought to be very clean and dry, otherwise it will not adhere. Glazier's putty is very similar to this.

**Roman Cement.**—This must be kept in well-closed vessels, and not moistened until the instant it is required for use.

**Plaster of Paris.**—This is mixed with water, milk, or weak glue, or starch water.

These three lutes stand a dull red heat: the two latter may be rendered perfectly impermeable to gaseous bodies by being smeared over with oil, or a mixture of oil and wax.

**Linseed or Almond Meal**, mixed to the consistence of a paste with water, milk, lime-water, or starch paste. This lute is very manageable and impermeable, but does not withstand a heat greater than about 500° F.

**Lime and Egg Lute.**—If just the sufficient quantity of water be added to quick lime to reduce it to a dry powder and that is mixed well and rapidly with white of egg diluted with its own volume of water, and the mixture spread immediately on strips of linen and applied to the part, then powered with quick lime, it forms a good cement. Instead of white of egg, lime and cheese may be used, or lime with weak glue water. This lute dries very rapidly, becoming very hard and adhering strongly to glass; but its great inconvenience is the want of flexibility.

**White Lead mixed with Oil.**—If this mixture be spread upon strips of linen, or bundles of tow, it acts much in the same manner as the lime lutes.

**Yellow Wax** is often used as a lute, but it becomes very brittle at a low temperature. It may be rendered less brittle, and at the same time more fusible, by an admixture of one-eighth crude turpentine.

**Soft Cement** is prepared by fusing yellow wax with half its weight of crude turpentine and a little Venetian red in order to colour it. It is very flexible, and takes any desired form under the pressure of the fingers.

**Waterproof Cement.**—Mr. Edmund Davy, F.R.S., has described a cement made by melting in a saucepan, two parts by weight of common pitch, and adding to it one part by weight of gutta percha, stirring and mixing them well together until they were completely incorporated with or united with each other. The mixture then formed a homogeneous fluid which may be used in this state for many purposes, and is remarkable on account of the facility and tenacity with which it adheres to metals, stones, glass. It may be poured into a large basin of cold water, in a thinner or thicker stream, or as a cake. In this state, while warm, it is quite soft, but may be soon taken up out of the water and drawn out into longer or pressed into shorter pieces, or cut or twisted into fragments, which may again be readily reunited by pressure. When the cement is cold, or before, it may be removed from the water and wiped dry, when it is fit for use. It is of a black colour; when cold, it is hard. It is not brittle, but has some degree of elasticity, which is increased by a slight increase of heat. It appears to be not so tough as gutta percha but more elastic. Its tenacity is very considerable, but inferior to gutta percha. It softens when put into water at about 100° F.; and if the heat is gradually increased it passes through intermediate states of softness, becomes viscous like bird-lime, and may be extended into threads of indefinite length: it remains in this state even when exposed for some time in a crucible, to the heat of boiling water, at 212° F.; when heated to above 100° F. it becomes a thin fluid. Water appears to have no other action upon it but that of softening it when warm or hot, and slowly hardening it when cold. The cement adheres strongly, if pressed on metal or other surfaces, though water be present, provided such surfaces be warm. This cement is applicable to many useful purposes. It adheres with great tenacity to metals, wood, stones, glass, porcelain, ivory, leather, parchment, paper, hair, feathers, silk, woollen, cotton, linen fabrics, &c. It is well adapted for glazing windows, or as a cement for aquariums. This cement does not appear to affect water, and it will apparently be found applicable for coating metal tanks;

to secure the joints of stone tanks; to make a glue for joining wood, which will not be affected by damp; and to prevent the depredations of insects on wood.

**Resinous, or Hard Cement,** is made by fusing together at the lowest possible temperature one part of yellow wax and five or six of resin, and then adding gradually one part of red ochre or finely-powered brickdust (plaster of Paris succeeds very well), and then raising the temperature to 212° at least until no more froth arises, or agitation takes places, and stirring it continually until cold. This cement is employed in a hot state. This lute is much used for fixing brass caps, &c., to air jars.

**Paper,** covered with common glue, is occasionally employed.

**Bladders,** cut in small strips, are occasionally used in covering other lutes, when the pressure of gas is considerable or when the lute is subject to strain from any other cause. They are digested in water until they become soft and flaccid; they are then applied to the part like a piece of pasted paper, by the pressure of the hand. These strips adhere very strongly to glass or earthenware, and their adhesive power may be much augmented by smearing them with white of egg. Lastly the joints made in this manner may be made firmer by binding them with string or fine wire.

**Caoutchouc.**—Tubes of vulcanised caoutchouc form a very ready means of attaching one piece of apparatus to another, and they possess the peculiar advantage of flexibility, which allows the various parts of the apparatus which they cannot connect to move in different directions to a slight extent, so that the whole is not so likely to be fractured as when connected in an inflexible manner. Caoutchouc is also less acted upon by gases and vapours than almost any other substance we know; even chlorine attacks it but slowly, and when unvulcanised it possesses the valuable property of forming a perfect joint when freshly-cut edges are brought and pressed together, hence the facility with which it is manufactured into tubes. The mode of manufacturing small connecting tubes, which are often required to be of unvulcanised caoutchouc, is as follows: Take a piece of the sheet caoutchouc of the required size, and warm it either in the hand or before a fire, until it is perfectly soft; then place it around a glass rod of the requisite size, pressing the edges close together with the fingers; when close together cut off the superabundance with a sharp pair of scissors, and the newly-cut edges will unite by simple pressure of the nail. When well executed the joint is scarcely apparent. In order to prevent the caoutchouc from adhering to the rods on which the tube is formed, a little moisture or dry starch may be employed. When caoutchouc is not at hand, oiled paper may be substituted, the joint being made of wax.

Faraday gives the following directions for luting iron, glass, or earthenware retorts, tubes, &c. for furnace operations. When the lute has to withstand a very high temperature it should be made of the best Stourbridge clay, which is to be made into a paste varying in thickness according to the opinion of the operator. The paste should be beaten until it is perfectly ductile and uniform, and a portion should then be flattened out into a cake of the required thickness, and of such a size as shall be most manageable with the vessel to be coated. If the vessel be a retort or flask, it should be placed in the middle of the cake, and the edges of the latter raised on all sides and gradually moulded and applied to the glass; if it be a tube if should be laid on one edge of the plate, and then applied by rolling the tube forward. In all cases the surface to be coated should be rubbed over with a piece of the lute dipped in water for the purpose of slightly moistening and leaving a little of the earth upon it; if any part of the surface becomes dry before the lute is applied it should be remoistened. The lute should be pressed and rubbed down upon the glass successively from the part where the contact was first made to the edges, for which purpose it is better to make them thin by pressure and also somewhat irregular in form, and if at all dry they should be moistened with a little soft lute. The general thickness may be about $\frac{1}{4}$ to $\frac{1}{3}$ of an inch.

Being thus luted, the vessels are afterwards to be placed in a warm situation, over the sand-bath or near the ash-pit, or in the sun's rays. They should not be allowed to dry rapidly or irregularly, and should be moved now and then to change their positions. To prevent cracking during desiccation, and the consequent separation of the coat from the vessel, some chemists recommend the introduction of fibrous substances into the lute, so as mechanically to increase the tenacity of its parts. Horse-dung, chopped hay and straw, horse and cow-hair, and tow cut short, are amongst the number. When these are used, they should be added in small quantity, and it is generally necessary to add more water than with simple lute, and employ more labour to ensure a uniform mixture. It is best to mix the chopped material with the clay before the water is put to it, and by adding the latter, to mix at first by stirring up the mass lightly with a pointed stick or fork; it will then be found easy, by a little management, to obtain a good mixture without making it very moist.

The luting ought to be made as dry as possible consistent with facility in working it. The wetter it is the more liable to crack in drying, and *vice versa*.

Mr. Willis recommends, when earthenware retorts, &c., are to be rendered impervious to air, the following coating: One ounce of borax is to be dissolved in half a pint of boiling water, and as much slaked lime added as will make a thin paste. This composition is to be spread over the vessel with a brush, and when dry, a coating of slaked lime and linseed oil is to be applied. This will dry sufficiently in a day or two, and is then fit for use.

**IRON CEMENT.**—This mixture is used for making permanent joints generally between surfaces of iron. Clean iron borings or turnings are to be slightly pounded, so as to be broken but not pulverised: the result is to be sifted coarsely, mixed with powdered sal-ammoniac and sulphur, and enough water to moisten the whole slightly. The proportions are 1 sulphur, 2 sal-ammoniac, and 80 iron. No more should be mixed than can be used at one time. Mr. Cooley states that he is informed by one of the first engineers in London that the strongest cement is made without sulphur, and with only one or two parts of sal-ammoniac to 100 of iron borings: but that when the work is required to dry rapidly, as for the steam joints of machinery wanted in haste, the quantity of sal-ammoniac is increased a little, and a very small quantity of sulphur is added. This addition makes it set quicker, but reduces its strength. Several excellent cements are described in Cooley's 'Cyclopædia of Practical Receipts.' From these the following are selected:—

**Beale's Cement.**—Chalk 60 parts: lime and salt, of each 20 parts; Barnsey sand 10 parts; iron filings or dust and blue or red clay of each, 5 parts. Grind together and calcine. This is patented as a fire-proof cement.

**Boiler Cement.**—Dried clay in powder 6 lbs., iron filings 1 lb., made into a paste with boiled linseed oil. This is used to stop leaks and cracks in iron boilers, stoves, &c.

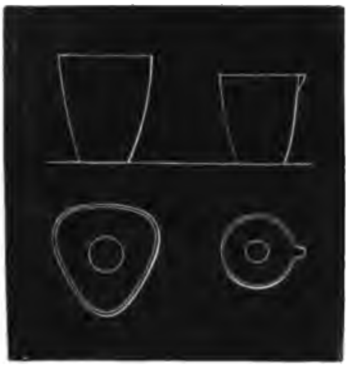
**Bruyere's Cement.**—Clay 3 parts; slaked lime 1 part: mix and expose them to a full red heat for 3 hours, then grind to powder.

This makes a good hydraulic cement.

**Oxychloride of Zinc Cement.**—In solution of chloride of zinc of 1·49 to 1·65 specific gravity, dissolve 3 per cent. of borax or sal-ammoniac, and then add oxide of zinc which has been heated to redness, until the mass is of a proper consistence. This cement becomes as hard as marble. It may be cast in moulds like plaster of Paris.

**Crucibles, Cupels, etc.**—The crucibles best known in commerce are the Hessian, the Cornish, the Stourbridge, and the London clay crucibles; charcoal, plumbago, iron, porcelain, platinum, silver and gold crucibles are also required in small operations. Of the clay crucibles, the London pots are much to be preferred on account of their very refractory nature. They resist the action of fused oxide of lead better than most clay crucibles, and they are also much better made than the two other kinds, being much smoother and more regularly formed. They have the form of a triangular pyramid (see fig. 58, crucibles and cover), and are made in such sizes that they fit one into the other, forming nests. The triangular form is very convenient, because there are three spouts, from either of which can be poured the fused contents of the pot. The Cornish crucibles are circular, and do not stand changes of temperature so well as the London pots, neither can they endure such an extreme of heat, for they agglutinate and run together at a temperature which does not touch the others. Dr. Percy says they are more generally useful than any other crucible. The Hessian pots are the worst of all; they do not stand moderate change of temperature without risk of fracture, so that they require to be very carefully used. There is also another kind of pot in use, made of the same material as the London crucibles, termed a 'skittle pot,' from its resemblance to the ordinary wooden skittle or ninepin. They are exceedingly useful for the fusion of large masses of matter, or such substances as boil or bubble much when heated. Plumbago or graphite crucibles are rapidly superseding all other kinds when metals have to be melted. They possess many advantages over clay crucibles. Their surface is very smooth; they are not liable to crack, however violent the changes of temperature may be to which they are subjected; they bear the highest heat without softening, and can be used repeatedly. Owing to the reducing property of the carbon they contain, they must not be employed when oxidising actions are required.

Fig. 58.



The Patent Plumbago Crucible Company have recently introduced a very excellent fluxing crucible. It is made of fine white china clay, is perfectly smooth inside and out, and will stand very high temperatures without softening.

Stourbridge clay crucibles are not much used. They require the greatest care in using them, and are spoilt after the first operation.

Porcelain crucibles are not used in large assaying or metallurgical operations, but they are invaluable in small laboratory experiments. They are practically infusible, are little liable to crack, and are almost unacted on by reagents and fluxes. In many cases they will replace the more expensive platinum crucibles, and where easily reducible metals are under treatment, they must be used in preference to platinum.

Crucibles in order to be perfect and capable of being used indifferently for any operation, ought to possess the four following qualities: firstly, not to break or split when exposed to sudden changes of temperature; secondly, to be infusible; thirdly, to be only slightly attacked by the fused substances they may contain; fourthly and lastly, to be impermeable, or nearly so, to liquids and gases. But as it is very difficult to unite all these qualifications, various kinds of pots are made to fulfil one or more of them.

In order to render crucibles capable of withstanding changes of temperature without breaking, a certain proportion of substances infusible by themselves, is mixed with the pasty clay; sand, flint, fragments of old crucibles, black-lead, and coke, are used for this purpose. They are reduced to a state of division more or less fine, according to the grain of the clay paste. For ordinary pots, the powder ought not to be very fine; but for porcelain crucibles it ought to be as fine as flour. The choice of these various bodies depends upon the use for which the crucible is intended.

The most refractory crucibles are those made with the pure clays, or such as contain little or no oxide of iron, and especially free from calcareous matters. Amongst those clays, the best are those which contain most silica; nevertheless, these are not absolutely infusible, and in the high temperature of a wind furnace they sometimes soften so much as actually to fall into a shapeless mass. This defect, as before stated, can be in some measure diminished by mixing with the clay a quantity of graphite or coke; either of these substances forms a kind of solid skeleton, which retains the softened clay, and prevents its falling out of shape.

Coke and black-lead are more efficacious than sand, because they have no action on clay, whilst sand forms a fusible compound with it. If too large a quantity of black-lead or coke be employed, it gradually consumes in the fire, and the pots become porous, and break at the least movement. Wood charcoal can be used instead of black-lead or coke, but is not so good, as it burns more readily.

Black-lead crucibles are generally composed of 1 part of refractory clay, and from 2 to 3 of black-lead. These pots withstand all possible changes of temperature without cracking, and their form is rarely changed by the heat, not because they are absolutely infusible, but because they are supported by the skeleton of graphite.

Crucibles into whose composition carbonaceous matters enter, reduce any oxides that may be heated in them, and hence are inconvenient in certain cases. They can, nevertheless, be employed in all cases by giving them a lining of clay, which must be tolerably thick, and well dried before use.

Earthen crucibles, which have not been baked at a white heat, are more or less permeable to liquids and gases, according to the grain. In order to render them impermeable to liquids, they must be heated to such a temperature as will suffice to fuse the outside. When treated in this way, however, they are very liable to crack with sudden changes of temperature: the best method, therefore, of rendering them capable of containing water, &c., is to coat them with the mixture of borax and lime described as Willis's lute.

In order that crucibles may resist the corrosive action of the fused substances contained within them, they must be as compact as possible, and the substance of which they are made must have little or no tendency to combine with the fused contents. The metals and their non-oxidised compounds neither attack clay nor black-lead; but there are, nevertheless, some metallic substances, galena, for instance, which, without exercising any chemical action on earthy matters, have the property of filtering through their pores.

The readily reducible oxides gradually corrode black-lead crucibles and those pots in the composition of which coke enters, by burning the carbonaceous matter. The greater number of these oxides, the alkalies, earths, and glasses (which are fusible silicates, borates, &c.), act more or less powerfully on the earthy base of all crucibles; so that these substances are most difficult to keep in fusion for any length of time. They attack the crucible layer by layer, dissolving the substance of which it is composed, and after a lapse of time rendering it so thin that it cannot withstand the pressure of the molten mass within it; and the fracture of the pot, and consequent loss of contents, is inevitable.

Under the same circumstances, all those crucibles whose texture is loose, are more readily corroded than those with a firm, compact body; because the corrosive substance filters to a certain depth in the former crucibles, and, in consequence, has a larger surface to act upon than when it is contained in a compact pot.

Earthen crucibles may be assayed by noticing the time they will contain fused litharge, which exercises a very corrosive action on them, honey-combing them in all directions; and those pots which contain it longest without undergoing much damage, may be considered the best. However, this method of assay is not exact, even by taking into account the thickness of the pot, for litharge runs through crucibles; firstly, because it is very fusible, and easily filters through their pores; and secondly, it has the property of forming fusible compounds with all the silicates by combining with them. From these remarks, it will be evident that a crucible whose grain is loose will readily allow litharge to pass through it, however slightly its substance may be fusible or acted on; or, on the contrary, it may be very easily acted on (even when infusible) when it has an extremely fine grain; so that the promptitude with which a crucible is traversed by litharge bears no relation to its fusibility. A crucible of pure quartz will be very readily attacked by litharge, because the latter has much affinity for silica, and the simple silicates of lead are all very fusible; whilst a crucible composed of silica, alumina, and lime, which by itself is very fusible, would be corroded less rapidly, because the oxide of lead has much less affinity for the earths than it has for the silica; moreover, it forms less fusible compounds with the earths than with silica alone. The assays of crucibles with litharge, if not of use in ascertaining their degree of fusibility, fulfils perfectly its object when it is wished to prove the resistance a crucible has to the corrosive action of various bodies in a state of fusion; for of all fusible substances, none exercise such a powerful action on earthy matters as litharge.

Crucibles ought not only to resist the corrosive action of those bodies they may contain, but also that of the ash produced by the combustion of the fuel in which they may be placed. These ashes being often calcareous, alkaline, or ferruginous, act on the clayey part of the crucibles exactly as the fluxes. Whence it follows, that those crucibles which contain litharge longest, will also resist the action of the fluxes best.

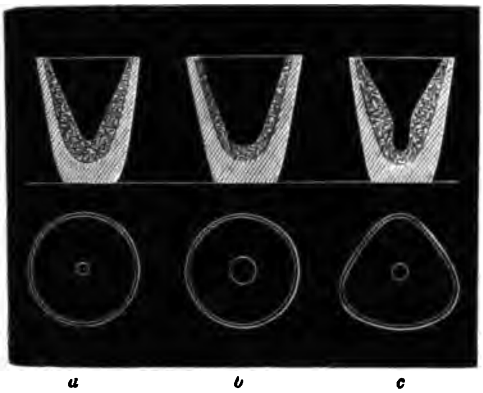
In order to ascertain the fusibility of a crucible, a direct experiment must be made, either by heating a piece in a crucible lined with charcoal, and scertaining if its angles be rounded, if its substance has become translucid, &c.; or, better still, by heating the crucible to be assayed with another whose properties are well known.

As to permeability, it may approximately be ascertained by filling two crucibles with water, and noting what length of time is required to empty them completely; the crucible which contains it longest being, of course, the least permeable.

To prove if a crucible be able to sustain great changes of temperature without breaking, introduce it, perfectly cold, into a furnace full of lighted coal: take it out when reddish white, and expose it to a current of cold air produced by a bellows or otherwise: if it stand these trials, it may be heated afresh and plunged red-hot into water, and if it be not broken, placed immediately in the fire. The best pots support all these operations without breaking; but it often happens that they are filled with innumerable small fissures, through which fused matters can pass. This can be ascertained by fusing rapidly in the assay pot a quantity of litharge: if these be present, the fused oxide will readily filter through them.

**Charcoal Crucibles.**—As all oxidised matters act readily on clay pots, and a great number of the metals and their compounds adhere to them, they have long since been replaced, under certain circumstances, by charcoal crucibles, which do not possess these disadvantages. The older assayers used merely a piece of charcoal, with a hole made in it, and then bound round with iron or other wire. The use of these has, however been abandoned for some time, and earthenware crucibles lined with charcoal have been substituted

FIG. 59.



(see fig. 59, *a*, *b*, and *c*). These may be considered as charcoal pots enveloped with refractory clay; they are solid, always free from cracks, and easy of preparation, and they have the same properties as the solid charcoal crucibles without their inconveniences.

In order to prepare these crucibles, the charcoal must be chosen carefully, so as to contain no foreign substances; it must be pulverised and passed through a sieve; the powder moistened with water, mixed with a spatula, and then kneaded with the fingers until it just adheres, and forms adhesive lumps without being sufficiently wet to adhere to the hand. Some advise the addition of gum to the water with which the charcoal is moistened. The crucible is moistened slightly by being plunged into water, and withdrawn as speedily as possible, and about half an inch in depth of the charcoal paste, prepared as above, placed in it; the paste is then pressed firmly down, by means of a wooden pestle: the blows are to be slight at first, and then increase in force until it is as firm as possible: another layer is then applied and pressed as before, and the process repeated until the crucible is quite full, taking great care to render all as firm as possible, especially at the sides. In order to make each layer adhere firmly to the other, the surface must be scratched rather deeply with the point of a knife before a new layer is applied. When the crucible is completely filled, a hole is to be scooped in the charcoal of about the form of the pot. The sides are then rendered smooth by friction with a glass rod. This is absolutely necessary, so that the metallic globules produced in an assay may not be retained by the asperities of the lining, but may be readily enabled to unite into one button. When a lined charcoal pot is well made, its sides are very smooth and shining. For ordinary use, the lining may be ⅜ths of an inch thick at the bottom and ⅛th or so at the sides; but in some cases, for instance, when the substance to be fused is capable of filtering through the lining and attacking the pot as a flux, it must be at least twice the above thickness in every part.

Lined crucibles have many advantages over plain crucibles. The lining gives them greater solidity, and prevents a loss of shape when softened; for plain crucibles are always three-fourths empty when their contents are fused, on account of contraction in volume: the pots then have nothing to sustain their sides when they soften towards the end of the assay, at which period the highest temperature is employed. Besides, vitreous matters do not penetrate the lining, and, exercising no action on it, can be obtained in a state of purity, and the exact weight determined: if fused in a plain pot, the weight could not be ascertained, because a portion would adhere to the sides, and the resulting mass would not be pure, having taken up a portion of the crucible in which the fusion was effected.

The lining, too, effects the reduction of certain metallic oxides by cementation, and does away with the necessity of adding powdered charcoal to the body to be reduced. This property is very valuable, because, when an oxide is reduced by mixing it with charcoal, an excess must always be employed, and this excess remains with the metal, and prevents its exact weight from being ascertained. No oxidising substances or bodies which readily part with oxygen (oxide of copper, for example) must be calcined in a plumbago or charcoal lined crucible, unless indeed the chemical union of the charcoal with the oxygen is desired.

**Lime Crucibles.**—Some years ago Deville proposed the use of crucibles cut out of solid blocks of pure lime, in order to prevent the introduction of carbon and silicon into metals and alloys during the process of fusion.

The results of experiments made with such crucibles were found to be extremely satisfactory, and metals fused therein, as iron, manganese, nickel, cobalt, &c., were obtained far purer and more malleable and ductile than when fused in the usual clay or brasque crucibles. Unless, however, the crucibles required were of very small size, it was found difficult to obtain blocks of lime for shaping them sufficiently large and free from flaws; and experiment showed a considerable loss, both by breakage when shaping them, and by their cracking when in the furnace. In order to obviate this, trials were made with clay crucibles lined with lime, but ineffectually, as these crucibles invariably melted down before the requisite heat was arrived at—a result due to the action of the lime itself upon the clay outer crucible.

Mr. David Forbes, F.R.S., has published in the 'Chemical News,' the result of some very valuable experiments on this subject. The arrangement he proposes fully answers the purpose; the crucibles being capable of standing the heat of melted wrought iron or cobalt without fusion or cracking, as well as of being made of any reasonable size.

A clay crucible of somewhat larger capacity than the desired lime one, is filled with common lamp-black, compressing the same by stamping it well down. The centre is then cut out with a knife until a shell or lining of lamp-black is left firmly adherent to the sides of the crucible, and about half an inch or less in thickness, according to the size of the crucibles; this lining is now well rubbed down with a thick glass-rod until its surface takes a fine glaze or polish, and the whole cavity is then filled up with finely-powdered caustic lime, thoroughly pressed down, and a central cavity cut out as before; or the lime-powder may be at once rammed down round a central core of the dimensions of the intended lime crucible.

This lime lining is naturally rather soft before being placed in the furnace, but upon heating, agglutinates, and forms a strong and compact crucible, which is prevented acting upon the outer one by the interposed thin lamp-black layer, and at the end of the experiment generally turns out as solid and compact as those made in the lathe.

Experiments made with such crucibles, even up to dimensions containing several pounds of metal, have proved them extremely well suited for these operations, and doubtless similar crucibles could be made, lined with magnesia or alumina as required. In some cases ordinary black-lead crucibles, lined with powdered lime, magnesia, or alumina, might possibly be found to answer.

Having frequently used lime crucibles in metallurgical operations, and having met with the inconvenience pointed out by Mr. Forbes, the writer can appreciate the great value of his improvement. It is one which cannot fail to be extensively adopted in metallurgical laboratories.

In certain particular experiments, crucibles are lined with other bodies besides charcoal and lime, such as silica, alumina, magnesia, or chalk, by merely moistening their respective powders with water, and applying the paste as above decribed for the charcoal. A slight layer of chalk lessens the liability of attack from fused litharge.

**Alumina Crucibles** are strongly to be recommended in many metallurgical operations. They are made in the following manner. Ammonia alum is ignited at a full red or white heat, when it leaves behind pure alumina in a dense compact form: this is to be finely powdered. To a solution of another portion of ammonia alum in water, ammonia is added, when alumina is precipitated in a gelatinous state: this is to be washed until free from sulphate of ammonia. The dense alumina is then mixed with water and worked up into a paste, the precipitated gelatinous alumina being kneaded in from time to time; this gives coherency: and when sufficient has been added (which must be ascertained experimentally), the mass may be moulded into shape. These crucibles require slow and careful drying; but they well repay all the care which is bestowed on them, for they do not readily crack, are attacked by very few fluxes, give out no impurities to metals which are melted in them, and are infusible at the highest heat of the furnace.

**Malleable Iron** crucibles are often very serviceable in assays of fusibility, and of certain selenides and sulphides, as in assays of galena or ordinary lead ore. They are either made of hammered sheet iron or by plugging up small iron tubes, as gun-barrels, &c. The latter are preferable, because thick solid crucibles can be used a number of times, whilst the others are necessarily very thin and can be used only once. Whenever iron crucibles are employed at a very high temperature, they must be placed in those of earthenware, which protect them from the oxidating action of the air; but when they are not heated above the temperature of a copper assay, they may be used naked, if tolerably thick.

For assays at the above temperature, cast-iron crucibles may be employed with advantage, instead of wrought-iron, because they are very nearly as good, and much less expensive.

**Platinum Crucibles.**—Platinum crucibles are invaluable in a laboratory. Few pieces of apparatus are used so frequently by the chemist. Their chief use is in the ignition of precipitates, and the decomposition of siliceous minerals by fusion with alkaline carbonates. They are preferable to porcelain, as not being fragile and being more readily heated to redness over the gas or spirit flame. Their most convenient size is 1½ inch high and 1¼ inch wide at the top.

In employing a crucible for the incineration of filters in quantitative assays by the wet way, it sometimes happens (as, for instance, with chloride of silver or sulphate of lead) that the employment of platinum is inadmissible. In these cases thin porcelain crucibles must be used. The analyst will, however, frequently experience difficulty, owing to the extreme slowness with which, in many cases, the last portions of the carbon of a filter are consumed when ignited in a porcelain crucible. It does not appear, however, that the following simple method of obviating the difficulty—as practised in the laboratory of Professor Scheerer, in Freiberg—has ever received the publicity which it deserves. Whenever a filter upon which a substance capable of injuring platinum has been collected has to be incinerated, the porcelain crucible or capsule in which the process is to be conducted, should be placed within a vessel of platinum of similar form, and the whole ignited in the usual way. Whether the greatly accelerated rapidity of combustion of the carbon which ensues depends upon a more equal distribution of heat brought about by the greater conducting power of the metal—an explanation which is current for the somewhat analogous case of copper-coated glass flasks—or whether, as seems probable, the power of the porcelain vessel to absorb heat be really increased by the interposition of the platinum; whether both these causes be of influence, or the result depends upon another less apparent reason; or finally, whether vessels of some other metal would not be preferable to those of platinum, are questions which are open to discussion.

Fresenius gives the following excellent directions as to the preservation of platinum crucibles. The analyst should acquire the habit of cleaning and polishing the platinum crucible always after using it. This should be done, as recommended by Berzelius, by friction with moist sea-sand, whose grains are all round, and do not scratch. The writer has found this method to answer extremely well. The sand is rubbed on with the finger, and the desired effect is produced in a few minutes. The adoption of this habit is attended with the pleasure of always working with a bright crucible, and the profit of prolonging its existence. This mode of cleaning is all the more necessary, when one ignites over gas-lamps, since at this high temperature crucibles soon acquire a grey coating, which arises from a superficial loosening of the platinum. A little scouring with sea-sand readily removes the appearance in question, without causing any notable diminution of the weight of the crucible.

In connection with some sensible remarks upon the above-mentioned use of sand in cleaning platinum crucibles, Erdmann explains in the following way the cause of the grey coating which forms upon platinum crucibles whenever they are ignited in the flame of Bunsen's gas-burner. This coating has given rise to much annoyance and solicitude among chemists. Indeed it has often been asserted that the use of Bunsen's burner is unadvisable in quantitative analysis, since by means of it the weight of platinum crucibles is altered and the crucibles themselves injured. The coating is produced most rapidly when the crucible is placed in the inner cone of the flame, and the more readily in proportion as the pressure under which the gas is burned is higher. Having found it advantageous to maintain, by means of a special small gas-holder, a pressure of four or five inches upon the gas used in his own laboratory, Erdmann observed that the strong gas-flame thus afforded, immediately occasioned the formation of a dull ring upon the polished metal placed in the inner flame, this ring being especially conspicuous when the crucible becomes red-hot; it increased continually, so that after long-continued ignition, the whole of the bottom of the crucible was found to be grey and with its lustre dimmed.

This ring is caused neither by sulphur, as some have believed, nor by a coating of inorganic matter, but is simply a superficial loosening of the texture of the platinum, in consequence of the strong heat; whence it first of all appears in the hottest part of the flame. In consequence of the serious damage which the gas furnace causes, many chemists now discard gas and ignite platinum crucibles over specially constructed spirit lamps.

In conjunction with Pettenkofer, Erdmann instituted several experiments, which have left but little doubt that the phenomenon depends upon a molecular alteration of the surface of the metal. If a weighed polished crucible be ignited for a long time over Bunsen's lamp, the position of the crucible being changed from time to time, in order that the greatest possible portion of its surface shall be covered with the grey coating, and its weight be then determined anew, it will be found that this has not increased. The coating cannot be removed either by melting with bisulphate of potash or with carbonate of soda. It disappears, however, when the metal is polished with sand; the loss of weight which the crucible undergoes being exceedingly insignificant, a crucible weighing 25 grammes having lost hardly half a milligramme. When the grey coating of the crucible is examined under the microscope, it may be clearly seen that the metal has acquired a rough, almost warty, surface, which disappears when it is polished with sand. Platinum wires, which are frequently ignited in the gas-flame—for example, the triangles which are used to support crucibles—become, as it is known, grey and brittle. Under the microscope, they exhibit a multitude of fine longitudinal cracks, which, as the original superficial alteration penetrates deeper, become more open, or, as it were, spongy, until, finally, the wire breaks.

If such wire is strongly and perseveringly rubbed with sand, the cracks disappear, and the wire becomes smooth and polished; for the grains of sand, acting like burnishers, restore the original tenacity of the metal, very little of its substance being rubbed off meanwhile. The loosening effect of a strong heat upon metals is beautifully exhibited when silver is ignited in the gas-flame; a thick polished sheet of silver immediately becoming dull white when thus heated. Under the microscope the metal appears swollen and warty. Where it has been exposed to the action of the inner flame along its circumference, this warty condition is visible to the naked eye. A stroke with the burnishing stone, however, presses down the loosened particles, and reproduces the original polish. This peculiar condition which the surface of silver assumes when it is ignited, is well known to silversmiths; it cannot be replaced by any etching with acids, and it must be remembered that what is dull white in silver, appears grey in platinum.

If each commencement of this loosening is again destroyed, the crucibles will be preserved unaltered, otherwise they must gradually become brittle. Crucibles of the alloy of platinum and iridium are altered like those of platinum, when they are ignited. It is, however, somewhat more difficult to reproduce the original polish of the metal by means of sand, as might be expected, from the greater hardness of the alloy.

The sand used should be well worn. When examined under the microscope, no grain of it should exhibit sharp edges or corners; all the angles should be obtuse.

If there are spots on the platinum crucibles which cannot be removed by the sand without wearing away too much of the metal, a little bisulphate of potassa is fused in the crucible, the fluid mass shaken about inside, allowed to cool, and the crucible finally boiled with water. There are two ways of cleaning crucibles soiled outside: either the crucible is placed in a larger one, and the interspace filled with bisulphate of potassa, which is then heated to fusion; or the crucible is placed on a platinum wire triangle heated to redness, and then sprinkled over with powdered bisulphate of potassa. Instead of the bisulphate borax may be used. Never forget at last to polish the crucible with sea-sand again.

When the crucible is clean, it is placed upon a clear platinum-wire triangle, ignited, allowed to cool in the desiccator, and weighed. This operation, though not indispensable, is still always advisable, that the weighing of the empty and the filled crucible may be performed under as nearly as possible the same circumstances.

In using platinum crucibles, it must be remembered that certain substances must not be ignited in them. Berzelius says, that 'it is improper to ignite in platinum vessels the caustic alkalies or the nitrates of any alkaline base, such as lime, baryta, or strontia, because the affinity of the alkali for oxide of platinum, causes a very considerable oxidation of the metal; and after the saline matter is removed, the surface of the metal is found to be honeycombed.'

The alkaline sulphides or the alkaline sulphates mixed with charcoal are inadmissible, because the sulphides so formed attack platinum even more energetically than the caustic alkalies: so are metals whose fusing-point is lower than that of platinum, because an alloy would be formed. Gold, silver, and copper, may be heated to dull redness in platinum vessels without danger; but fused lead cannot come in contact with platinum without destroying it. A drop of fused lead, tin, zinc, or bismuth, placed on red-hot platinum, always produces a hole. Neither can a phosphide or phosphoric acid mixed with charcoal be ignited in vessels of platinum, because a phosphide of platinum is produced, which is an exceedingly brittle compound.

In analyses by the humid method, nitro-hydrochloric acid (*aqua regia*), even when very dilute, must not be allowed to come in contact with platinum, and, as a general rule, liquids containing either free chlorine, bromine, or iodine, must not be boiled in platinum capsules.

**Cupels.**—These are vessels in which the operation termed cupellation is carried on. They must be made of such substances as are not acted upon by certain fused oxides, as those of lead or bismuth, and their texture has to be sufficiently loose to allow of the oxides penetrating their substance readily, and yet be sufficiently strong to bear handling without breaking.

There are several substances of which cupels might be made, which will fulfil all these conditions, but only one is in general use, viz. the ash of burnt bones. This consists principally of phosphate of lime, with a little carbonate and some fluoride of calcium. Berzelius found that bones of oxen contained 57 parts of phosphate of lime for every 3·8 parts of carbonate of lime, whilst, according to Barros, sheep bones contain 80 parts of phosphate of lime to 19 parts of carbonate of lime. When bones are burnt whole they likewise contain mineral matter derived from the cartilage, such as alkaline sulphates and carbonates. The greater part of the carbonate of lime is likewise converted into caustic lime.

The bones of sheep and horses are best for cupels. In getting rid of the organic matter, it is advisable to boil them repeatedly in water before burning them. This dissolves a great part of the organic matter. If the bones are not rendered quite white by the first ignition, but contain a little carbon, they should be ground up, moulded into shape, and burned again.

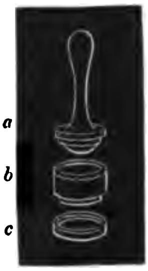
Care should be taken not to heat the bone earth too strongly. In this case the bones will have a smooth, glassy fracture, and will not be sufficiently spongy or absorbent to make good cupels.

When the bones are burnt white throughout, they must be finely ground, sifted, and washed several times with boiling distilled water till all soluble salts are removed. The finest particles of the powdered bone earth will remain longest suspended in the washing waters. This must be allowed to settle separately, and should be reserved for giving a final coating to the surface of the cupels; this coating acts, to a certain extent, like a fine filter, and may be applied to all cupels, although the body of the cupel is made of different materials.

For the body of the cupels, the bone-ash should be about as fine as wheat flour. If too coarse, litharge containing silver will be absorbed into its pores, and will occasion a loss of silver.

Cupels must neither crack nor alter in texture at a white heat. It is very important that they should not contain carbon, and therefore, in making them, the bone-earth must not, as sometimes recommended, be mixed with beer, or water containing adhesive substances. Nothing but pure water should be used, and the mixture should be just sufficiently moist to adhere strongly when well pressed, but not so moist as to adhere to the finger or the mould employed to fashion the cupels. The mould (fig. 60) consists of three pieces; one a ring, *b*, having a conical opening; another a pestle, *a*, having a hemispherical end fitting the larger opening of the ring; and the third, *c*, a piece of turned metal, into which *b* fits; *c* serves to form an even bottom to the cupel. In order to mould the cupels, proceed as follows: Place the ring on the lower piece *c*, and fill it with the composition; then place the pestle upon it, and force it down as much as possible: by this means the moistened bone-ash will become hardened, and take the form of the pestle; the latter must then be driven as much as possible by repeated blows from a hammer, until quite home. The surface of the cupel may then have sifted over it a little of the very fine levigated bone-ash, and the pestle hammered again on it. It is then to be turned lightly round, so as to smooth the inner surface of the cupel, and withdrawn: the cupel is removed from the mould by a gentle pressure on the narrowest end. When in this state, the cupel must

FIG. 60.



be dried gently by a stove; and lastly, ignited in a muffle, to expel all moisture. It is then ready for use.

There are two or three points to attend to in manufacturing the best cupels. Firstly, the powdered bone-ash must be of a certain degree of fineness; secondly, the paste must be neither too soft nor too dry; and thirdly, the pressure must be made with a certain degree of force. A coarse powder, only slightly moistened and compressed, furnishes cupels which are very porous, break on the least pressure, and, as before mentioned, allow small globules of metal to enter into their pores.

When, on the contrary, the powder is very fine, the paste moist, and compressed strongly, the cupels have much solidity, and are less porous; the fine metal cannot penetrate them, but the operation proceeds very slowly: besides, the assay is likely to become dulled, and incapable of proceeding without a much higher degree of temperature being employed.

Cupels for assaying silver bullion are sometimes made of equal parts of bone-ash and beechwood-ash; and for assaying gold, 2 parts of beechwood-ash, and 1 part of bone-ash are used. The hemispherical cavity of both these kinds are coated with the fine levigated powder of bone-ash.

Beechwood-ash is preferred for cupels on account of the larger proportion of phosphoric acid it contains.

According to Hertwig, beechwood-ash contains in 100 parts:—

| | |
|---|---|
| Carbonate of potash | 11·72 |
| Carbonate of soda | 12·37 |
| Sulphate of potash | 3·49 |
| Carbonate of lime | 49·54 |
| Carbonate of magnesia | 7·74 |
| Phosphate of lime | 3·32 |
| " magnesia | 2·92 |
| " iron | 0·76 |
| " alumina | 1·51 |
| " manganese | 1·59 |
| Silica | 2·46 |

**Scorifiers.**—A scorifier (fig. 61) is a vessel made much in the shape of a cupel, but of crucible earth. The proper use both of cupels and scorifiers will be explained under the head of silver assay.

**Methods of Measuring the Heat of Furnaces.**—As much of the accuracy of an assay depends on the temperature at which it is made, and as the temperature required varies with each metal, it is very desirable to possess some means of ascertaining the heat of the furnace more accurately than by the eye. Many persons have devised instruments, called pyrometers, for this purpose; the earliest being those of Mr. Wedgwood and the late Professor Daniell, of King's College.

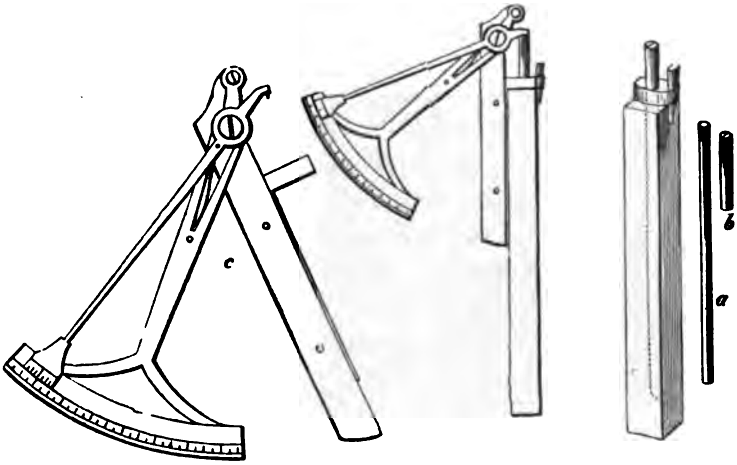
We shall not give a description of Wedgwood's pyrometer, as although very ingenious and useful in the absence of a better instrument, it has long gone out of use.

Fig. 61.

Its indications are very inaccurate, from the fact that the clay cylinders, whose contraction serves to measure the temperature, will contract as much by the long continuance of a low heat as by the short continuance of a high one. Hence the degrees of heat measured by Wedgwood's pyrometer have been enormously exaggerated. It was long since noticed that it did not produce comparable effects; and this was supposed to proceed wholly from the impossibility of obtaining clay perfectly alike for each experiment.

Daniell's pyrometer is composed of a rod of platinum simply laid in a groove made of refractory clay, and baked in the highest degree of heat. This rod rests at one end on the edge which terminates the groove, and at the other on a lever with two arms, the larger of which forms a needle on a graduated arc of a circle; so that the removal of this needle from its position marks the additional length which the metal acquires by the heat. The following is Daniell's description of his pyrometer: 'It consists of two parts (see fig. 62), which may be distinguished as the register and the scale. The register is a solid bar of black-lead or earthenware highly baked. In this a hole is drilled, into which a bar of any metal, *a*, six inches long, may be dropped, and which will then rest upon its solid end. A cylindrical piece of porcelain, *b*, called the index, is then placed upon the top of the bar, and confined in its place by a ring or strap of platinum passing round the top of the register, which is partly cut away at the top, and tightened by a wedge of porcelain. When such an arrangement is exposed to a high temperature, it is obvious that the expansion of the metallic bar will force the index forward to the amount of the excess of its expansion over that of the black-lead, and that when again cooled it will be left at the point of greatest elongation. What is now required is the measurement of the distance which the index has been thrust forward from its first position, and this, though in any case but small, may be effected with great precision by means of the scale, *c*.[*]

FIG. 62.



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[*] Daniell's *Chemical Philosophy*, p. 111.

This is independent of the register, and consists of two rules of brass accurately joined together at a right angle by their edges, and fitting square upon the two sides of the black-lead bar. At one end of this double rule, a small plate of brass projects at a right angle, which may be brought down upon the shoulder of the register formed by the notch cut away for the reception of the index. A movable arm is attached to this frame, turning at its fixed extremity on a centre, and at its other carrying the arc of a circle, whose radius is exactly five inches, accurately divided into degrees, and thirds of a degree. Upon this arm, at the centre of the circle, another lighter arm is made to turn, one end of which carries a nonius with it, which moves upon the face of the arc, and subdivides the former graduation into minutes of a degree; the other end crosses the centre, and terminates in an obtuse steel point, turned inwards at a right angle. When an observation is to be made, a bar of platinum or malleable iron is placed in the cavity of the register; the index is to be pressed down upon it, and firmly fixed in its place by the platinum strap and porcelain wedge. The scale is then to be applied by carefully adjusting the brass rule to the sides of the register, and fixing it by pressing the cross piece upon the shoulder, and placing the movable arm so that the steel part of the radius may drop into a small cavity made for its reception, and coinciding with the axis of the metallic bar. The minute of the degree must then be noted which the nonius indicates upon the arc. A similar observation must be made after the register has been exposed to the increased temperature which it is designed to measure, and again cooled, and it will be found that the nonius has been moved forward a certain number of degrees or minutes. The scale of this pyrometer is readily connected with that of the thermometer by immersing the register in boiling mercury, whose temperature is as constant as that of boiling water, and has been accurately determined by the thermometer. The amount of expansion for a known number of degrees is thus determined, and the value of all other expansions may be considered as proportionate.

By Daniell's pyrometer the melting point of cast iron has been ascertained to be 2786°, and the highest temperature of a good wind furnace, 3300° Fahrenheit—points which were estimated by Mr. Wedgwood at 17,977° and 21,877° respectively.

The following is a list of the melting points of some of the metals as ascertained by Professor Daniell; and it is obvious that in an assay of each particular metal the temperature employed must exceed by a considerable number of degrees its melting point. The table is, therefore, very useful.

| | Fahr. |
|---|---|
| Tin melts at | 422° |
| Cadmium | 442 |
| Bismuth | 497 |
| Lead | 612 |
| Zinc | 773 |
| Silver | 1860 |
| Copper | 1996 |
| Gold | 2016 |
| Cast iron | 2786 |

Cobalt and nickel rather less fusible than iron.

Mr. S. Wilson* has described an ingenious process of measuring high temperatures. He exposes a given weight of platinum or Stourbridge clay to the action of the heat which is to be measured, and then quenches it in a definite weight of water at a certain temperature. Thus, if the piece of platinum weigh 1,000 grains and the water 2,000 grains at 60° F., and should the heated platinum when dropped into the water raise its temperature to 90°, then, $90° - 60° = 30°$; which, multiplied by 2 (because the weight of the water is twice that of the platinum), gives 60°—the temperature to which a weight of water equal to the platinum would have been raised. To convert this into Fahrenheit degrees we must multiply by $31\frac{1}{4}$, which is the specific heat of water as compared with platinum, that of the latter being 1. Therefore, $60° \times 31\frac{1}{4} = 1875°$, which will be the temperature of the furnace.

This principle has been very well carried out by O. Byström, a captain in the Swedish artillery. His instrument is called the hydropyrometer. It consists of two parts, shown in fig. 63. A represents a ball of platinum or other metal or alloy, according to the supposed temperature. B is a vessel of water. The portion *a*, *a*, is of brass, with two holes in the upper part; one, *b*, *b*, for the *mixer*, and the other, *c*, *c*, for the mercurial thermometer. There is also another hole at the lower part, *d*, by which the water is emptied. *e*, *e*, is a wooden case well screwed together. The mixer C consists of a conical ring, *f*, and wires of brass, which connected with the lower part of the ring, form the small cage, *g*. The upper portion is prolonged into a funnel, and has attached to it a small handle, *h*, used to take hold of and turn the mixer. The thermometer *k*, surrounded with the case *l*, is graduated to show one-tenth of a degree Centigrade.

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* *Philosophical Magazine*, ser. iv. vol. iv. p. 157.

FIG. 63.

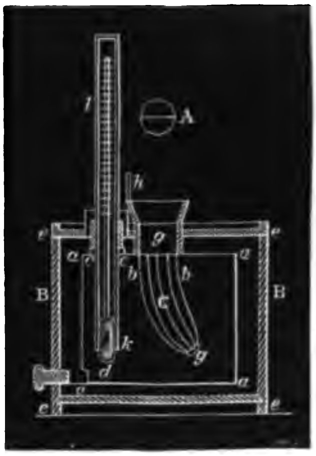
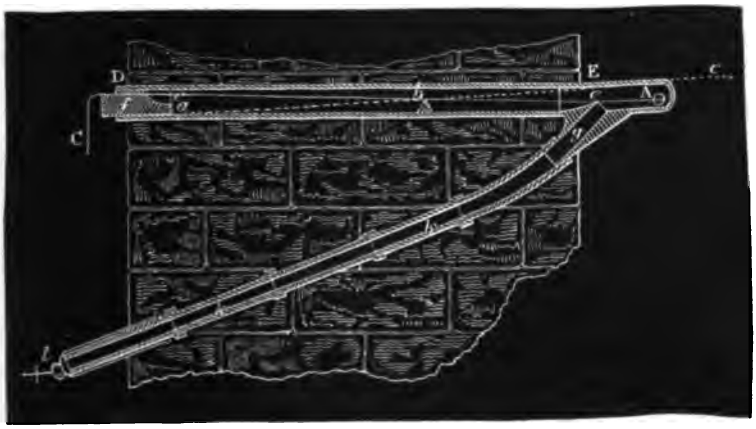


Fig. 64 shows how the instrument is arranged. Place the

FIG. 64.



ball A on the end of the rod C, which is then introduced and slid along the two points *a*, *b*, to the end of the muffle E, through the opening D. Then push the wedge-shaped stopper *f* into this opening, until the rod, which is balanced on the point *b*, touches the point *e*. Then close the mouth D with clay.

As soon as the ball has acquired the temperature of the furnace (in three or four minutes), draw out the rod. The ball touching the point *e* becomes detached and falls, rolling down the canal *g*, *k*, closed below by the valve *l*, when it falls into the vessel previously filled with a determined weight of water, B, through the funnel *f*. After having closed the funnel with a cork, turn the mixer very slowly two or three times, slightly shaking the instrument at the same time.

By taking the temperature of the water before ($t$) and after ($t'$) the operation, the difference ($t' - t$) is easily found. Reference is then made to the following table (see p. 137).

This table is calculated for the weights of 300 grammes of water, 7 grammes of steel, and 8 grammes of platinum. For each degree $t' - t$, a temperature $x$ is found to correspond, to which is added the final temperature $t'$.

EXAMPLE 1.

$t' = 17.^{\circ}$
$t = 14{\cdot}4$ steel
$t' - t = 2{\cdot}6$
$x$ the corresponding temperature $= 660^{\circ}$
$t'$ . . . . . . . $= 17$
the temperature of the furnace ($x + t'$) . . . . $= 677^{\circ}$

EXAMPLE 2.

$t' = 20.^{\circ}$
$t = 17{\cdot}75$ platinum
$t' - t = 2{\cdot}25$
$x$ the corresponding temperature . . . . $= 1550^{\circ}$
$t'$ . . . . . . $= 20$
the temperature of the furnace ($x + t'$) . . . $= 1570$

One or two other methods of measuring high temperatures applicable to special cases may here be mentioned.

Becquerel has proposed a very excellent plan for measuring high temperatures, by means of the thermo-electric current generated by heating the junction of two platinum wires of different diameters. In a similar manner, the thermo-electric current produced by heating two wires of platinum and palladium, melted together at one end, has been used as a pyrometer.

A good plan for comparing the temperatures of two furnaces is to prepare alloys of platinum and gold, containing definite quantities, say, 5, 10, 15, 20 % &c. of gold.

| $x$ | $t' - t$ | | $x$ | $t' - t$ | $x$ | $t' - t$ |
|---|---|---|---|---|---|---|
| Temperature centigrade | Steel | Platinum | Temperature centigrade | Platinum | Temperature centigrade | Platinum |
| 300 | 0·89 | 0·26 | 920 | 0·97 | 1520 | 2·17 |
| 50 | 1·08 | ·30 | 40 | 1·00 | 40 | ·22 |
| 400 | ·27 | ·35 | 50 | ·01 | 50 | ·24 |
| 50 | ·49 | ·40 | 60 | ·03 | 60 | ·27 |
| 500 | ·72 | ·45 | 80 | ·06 | 80 | ·32 |
| 50 | ·97 | ·50 | 1000 | ·09 | 1600 | ·38 |
| 600 | 2·24 | ·56 | 20 | ·12 | 20 | ·44 |
| 20 | ·36 | | 40 | ·15 | 40 | ·50 |
| 40 | ·48 | | 50 | ·16 | 50 | ·53 |
| 50 | ·54 | ·62 | 60 | ·18 | 60 | ·56 |
| 60 | ·60 | | 80 | ·21 | 80 | ·62 |
| 80 | ·73 | | 1100 | ·25 | 1700 | ·68 |
| 700 | ·86 | ·68 | 20 | ·29 | 20 | ·74 |
| 10 | ·92 | | 40 | ·33 | 40 | ·80 |
| 20 | ·98 | | 50 | ·35 | 50 | ·83 |
| 30 | 3·05 | | 60 | ·37 | 60 | ·86 |
| 40 | ·12 | | 80 | ·41 | 80 | ·92 |
| 50 | ·19 | ·74 | 1200 | ·45 | 1800 | ·99 |
| 60 | ·26 | | 20 | ·49 | 20 | 3·06 |
| 70 | ·34 | | 40 | ·53 | 40 | ·13 |
| 80 | ·42 | | 50 | ·55 | 50 | ·16 |
| 90 | ·50 | | 60 | ·57 | 60 | ·20 |
| 800 | ·58 | ·80 | 80 | ·61 | 80 | ·27 |
| 10 | ·66 | | 1300 | ·65 | 1900 | ·34 |
| 20 | ·74 | ·82 | 20 | ·69 | 20 | ·41 |
| 30 | ·82 | | 40 | ·73 | 40 | ·48 |
| 40 | ·90 | ·85 | 50 | ·75 | 50 | ·51 |
| 50 | ·98 | ·86 | 60 | ·77 | 60 | ·55 |
| 60 | 4·07 | ·88 | 80 | ·82 | 80 | ·62 |
| 70 | ·16 | | 1400 | ·87 | 2000 | ·70 |
| 80 | ·25 | ·91 | 20 | ·92 | | |
| 90 | ·34 | | 40 | ·97 | | |
| 900 | ·43 | ·94 | 50 | 1·99 | | |
| 5 | ·47 | | 60 | 2·02 | | |
| 10 | ·52 | | 80 | ·07 | | |
| 15 | ·57 | | 1500 | ·12 | | |

**Observation.**—$x + t'$ is the temperature of the furnace.

These fuse at intermediate temperatures between gold and platinum. By placing small angular chips of these alloys separately in muffles, and noticing which are melted, which softened only, and which resist the action of the heat, an idea of the power of the furnace is obtained. In this way the amount of heat required to perform any operation may be registered for future reference, by simply recording that it was sufficient just to melt, say a 20 gold 80 platinum alloy.

# CHAPTER V.

## FUEL: ITS ASSAY AND ANALYSIS.

**Before** treating of the assay of metals and metalliferous ores, it is advisable to devote some space to the important subject of fuel. The substances employed as fuel, although all of vegetable origin, are derived either from the vegetable kingdom (wood), or from the mineral kingdom (peat, brown coal, coal, anthracite). These natural fuels can be converted into artificial fuels by heating them more or less out of contact with the air (charcoal, turf-charcoal, coke).

The essential elements of combustible matters are carbon, oxygen, and hydrogen; nitrogen being present sometimes, but only in small proportions. These constitute the organic part; various salts and silica constitute the inorganic part, or ash. The valuable constituents of fuel, on which its calorific and reducing powers depend, are the carbon and hydrogen, and it is upon the combustion or union of these elements with oxygen to form carbonic acid and water, that the effect of the fuel depends.

The more oxygen a fuel contains the less carbon and combustible gases it will yield, and the more hydrogen, the more combustible gases.

| | | | |
|---|---|---|---|
| The proportion of hydrogen to oxygen in | wood . | is | 1 : 7 |
| " " " | turf . | " | 1 : 6 |
| " " " | fossil-wood | " | 1 : 4 |
| " " " | coal . | " | 1 : 2–3 |
| " " " | anthracite | " | 1 : 1 |

The more oxygen, the less carbon the fuel contains, thus:

| | | | |
|---|---|---|---|
| Anthracite | contains about | 90% | carbon |
| Coal | " " | 80 " | " |
| Brown coal | " " | 70 " | " |
| Fossil-wood and turf | " " | 60 " | " |
| Wood | " " | 50 " | " |

The more carbon a fuel contains, the greater heat it produces, and the more difficult it is to ignite.

The greater the amount of hydrogen in a fuel, the more inflammable it will be, and the larger flame it gives, the hydrogen being evolved below a red heat. But the more carbon present, the less flame. These differences are shown in a blazing fire and a glowing fire. In a flame the hottest part is at the periphery, whilst in a glowing fire the greatest heat is in the immediate contact of the burning surface.

The assay of fuel comprises the following examinations:

1. The examination of the external appearance of the fuel, its porosity or compactness, its fracture, the size and shape of the pieces composing it.
2. Determination of the adhering water.
3. The specific gravity.
4. Determination of the absolute heating power.
5. Determination of the specific heating power.
6. Determination of the pyrometric heating power.
7. Determination of the volatile products of carbonisation.
8. Examination of the coke or charcoal left behind on carbonisation, both with regard to quality and quantity.
9. Determination of the amount of ash, and its composition.
10. Determination of the amount of sulphur.
11. Examination of any other peculiarity which may be noticed during the burning or carbonisation of the fuel.

1. **External Appearance of the Fuel, its Porosity, Compactness, Fracture, Size, and Shape of Pieces.**—From the outward appearance of a fuel, its cleavage, and an examination of the embedded earthy matter, iron pyrites, gypsum, &c., its applicability to any special purpose may be judged. Its degree of inflammability, together with the pressure of blast which it will bear in the furnace, partly depends on the more or less compactness of the fuel. The amount of loss which it will suffer in transport depends upon its friability. Playfair and De la Beche * determined

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* Dingl. cx. 212, 263; cxiv. 346. Liebig's 'Jahresber.,' 1847–1848, p. 1117 1849, p. 708.

the amount of this loss in coal by rotating in a barrel different qualities of coal for the same time. The powder produced was separated and weighed, and in this way the friability or cohesion of a fuel could be expressed in percentages. Schrötter * made the same experiments with brown coal.

The size and form of the pieces composing the fuel is important, as on this depends the space occupied in its stowage—an important point for steam-vessels. This space cannot be calculated from its specific gravity, but must be ascertained by direct measurement. The space occupied will be smallest when the form of the lumps is cubical. Playfair and De la Beche have also investigated this subject.

2. **Determination of the Adhering Water.**—The water contained in a fuel exerts great influence on its heating power. It not only increases its bulk, but it acts injuriously by abstracting a certain quantity of heat required for its evaporation, and it also causes imperfect combustion. For this reason, wood, turf, and brown coal never give so high a temperature as coal, anthracite, and coke.

The determination of the adhering water is effected by drying a certain weight of the pounded fuel in a water-bath at 212° F. or in an air-bath at 220°. It may also be ascertained by placing a certain weight of the powdered fuel in a glass tube, heating to 212°, and passing over it air dried by means of chloride of calcium, till the fuel no longer loses weight. The amount of water which the dried fuel will absorb from the atmosphere in twenty-four hours, should also be determined, in order to ascertain its hygroscopic qualities.

3. **Determination of the Specific Gravity.**—The specific gravity of a fuel depends on its density and the amount of ash, and it appears also to be in proportion to its greater or less inflammability. Of two equal volumes of carbonised fuel, the one will produce the greatest heating effect which has the greatest specific gravity, provided the density is not produced by mineral constituents.

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* Wien. akad. Ber. 1849, Nov. and Dec. p. 240. Liebig's 'Jahresber.,' 1849, p. 709.

The determination of the specific gravity is difficult, and sometimes uncertain, owing to the cleavage of the fuel, and the entanglement of air in its pores. The best way of obviating this difficulty, is to finely powder the fuel before taking its specific gravity. Full directions will be found in a subsequent chapter.

4. **Determination of the Absolute Heating Power.**—The value of a fuel for any purpose depends chiefly on its price and the quantity required for that purpose. The *quantity* required depends on the heating power possessed by a certain *weight* of fuel (its *absolute* heating power) or that possessed by a certain *volume* (its *specific* heating power).

The less oxygen, ash, and water the fuel contains, the greater its heating power will be, and this will also increase in proportion to the carbon and hydrogen present.

Whether the combustion is effected quickly or slowly, the amount of heat produced will be the same, but the degree of temperature attained will be very different. This latter constitutes the *pyrometric* heating power.

The determination of the absolute heating power of a fuel may be effected,—

*a.* By heating a definite quantity of water from 32° F. to 212°;

*b.* By ascertaining how much fuel is required to melt a known weight of ice;

*c.* By ascertaining how much water may be evaporated by 1 lb. of different kinds of fuel;

*d.* By ascertaining how much the temperature of a room increases by burning a certain weight of a fuel in a stove.

*e.* By ascertaining the elementary composition of the fuel, and calculating how much oxygen will be required to convert the carbon and hydrogen into carbonic acid and water; the quantity of heat produced will be in proportion to the amount of oxygen consumed.

*f.* By Berthier's method,

*g.* By Dr. Ure's method.

According to Berthier, the most convenient method for ascertaining the comparative calorific power of any combustible matter is by means of litharge. He says: It has been proved by the experiments of many philosophers, that the quantities of heat emitted by combustible substances are exactly proportioned to the amounts of oxygen required for their complete combustion. Whence, after the elementary constitution of any combustible is known, its calorific power is easily determined by calculation. For instance, it is only necessary to ascertain the quantity of oxygen absorbed in the conversion of all its carbon into carbonic acid, and all its hydrogen into water, and compare that quantity with that which is consumed in burning a fuel whose calorific power is well ascertained. Such a substance is charcoal.

By adopting the principle just pointed out, it may be conceived that, without knowing the composition of a fuel, its heating power may be ascertained by determining the amount of oxygen it absorbs in burning. This can be done in a very simple and expeditious manner, if not exactly, at least with sufficient exactitude to afford very useful results in practice. It is as follows: many metallic oxides are reduced with such facility that when heated with a combustible body, the latter burns completely, without any of its elements escaping the action of the oxygen of the oxide, if the operation be suitably performed. The composition of the oxide being well known, if the weight of the part reduced to the metallic state be taken, the quantity of oxygen employed in the combustion can be ascertained. In order to collect the metal and separate it from the non-reduced mass, it must be fusible as well as its oxide. Litharge fulfils these conditions, and experiment has proved that it completely burns the greater part of all ordinary fuels; the only exceptions are some very bituminous matters containing a large proportion of volatile elements, a portion of which escapes before the temperature is sufficiently high to allow the reduction to take place. The experiment is made as follows:—10 grains of the finely-powdered, or otherwise divided fuel is mixed with about 400 grains of litharge. The mixture is carefully placed in an earthen crucible, and covered with 200 grains more litharge. The crucible is then placed in the fire and gradually heated. When the fusion is perfect, the heat is urged for about ten minutes, in order that all the lead may collect into a single button. The crucible is then taken from the fire, cooled, broken, and the button of lead weighed. Sometimes the button is livid, leafy, and only slightly ductile; in which case it has absorbed a little litharge. This can be partially prevented by fusing slowly, and adding a little borax.

Two assays, at least, ought to be made, and those results which differ more than a grain or two ought not to be relied on. The purer the litharge, the better the result; it ought to contain as little minium as possible. It is an excellent plan to mix up the litharge of commerce with one or two thousandths of its weight of charcoal, and fuse the whole in a pot; when cold, pulverise the litharge, which will now be deprived of minium.

Pure carbon produces, with pure litharge, thirty-four times its weight of lead, and hydrogen 103·7 times its weight of lead; that is to say, a little more than three times as much as carbon. We can, therefore, from these data, find the equivalent of any fuel, either in carbon or hydrogen.

When a fuel contains volatile matters, the quantity can be ascertained, as before pointed out, by ignition in a close tube or crucible. If, further, we ascertain the proportion of lead it gives with litharge, it is easy to calculate the equivalent in carbon of the volatile matters, and, in consequence, to ascertain its calorific value.

Supposing that a substance gives by distillation C parts of coke, or carbon, having deducted the weight of the ash and of volatile substances, and that it produces P parts of lead with litharge. The quantity C of carbon would give $34 \times C$ of lead; the quantity of volatile matter would give but $P - 34 \times C$; it would be equivalent to $\frac{P - 34 \times C}{34}$ of carbon: whence it follows that the quantity of heat developed by the charcoal, the volatile matter, and the unaltered combustible, will be to each other as the numbers $34 \times C$, $P - 34 \times C$, and P.

Dr. Ure[*] says, speaking of the above method of assay, 'On subjecting this theory to the touchstone of experiment, I have found it to be entirely fallacious. Having mixed very intimately 10 grains of recently calcined charcoal with 1000 parts of litharge, both in fine powder, I placed the mixture in a crucible, which was so carefully covered as to be protected from all fuliginous fumes, and exposed it to distinct ignition.

'No less than 603 grains of lead were obtained, whereas, by Berthier's rule, only 340 or 346·6 were possible. On igniting a mixture of 10 grains of pulverised anthracite with 500 grains of pure litharge previously fused and pulverised, I obtained 380 grains of metallic lead. In a second experiment, with the same anthracite and the same litharge, I obtained 450 grains of lead; and in a third, only 350 grains. It is therefore obvious that this method of Berthier's is altogether nugatory for ascertaining the quantity of carbon in coals, and is worse than useless in judging of the calorific qualities of different kinds of fuel.'

This discrepancy in the results obtained by Dr. Ure is very perplexing, and does not at all accord with Berthier's experience, as shown by his experiments, or by the author's, on the subject. The latter never had a difference of more than 50 grains, and in general only two or three, which latter result is satisfactory. The only precaution he found necessary was to heat very gradually until the mixture was fully fused, and then to increase the fire to bright redness for a few minutes.

Further experiments have been made by the author on this subject, and he has succeeded most perfectly in estimating the value of a fuel. With the litharge of commerce, which contains much minium, the process is never exact: results have been obtained differing as much as 40 or 50 grains when the litharge employed had not been purified, and to purify it completely is a troublesome process. This difficulty may be completely obviated, however, by substituting for litharge, white lead, using for each 10 grains of fuel 700 grains of white lead, which are well mixed with it, and 300 grains of pure white lead to cover the mixture. When the whole is heated, the carbonate of lead decomposes, forming pure oxide of lead, which is then reduced, as in the former case. By this process the results correspond to 1 grain in the quantity of lead produced from a given sample of fuel. Of course great care must be taken that the white lead is genuine.

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[*] 'Supplement to the Dictionary of Arts, Mines, and Manufactures.'

Commercial samples are frequently adulterated with sulphate of lead and sulphate of baryta, oxychloride of lead, oxide of zinc, &c. This is a serious drawback to this otherwise excellent modification.

The following is the method of ascertaining the calorific power of fuel, employed by Dr. Ure, and described in his 'Supplement.'

'The following calorimeter, founded upon the same principle as that of Count Rumford, but with certain improvements, may be considered as an equally correct instrument for measuring heat with any of the preceding (Lavoisier, Meyers, and others), but one of much more general application, since it can determine the quantity of heat disengaged in combustion, as well as the latent heat of steam and other vapours.

'It consists of a large copper bath capable of holding 100 gallons of water. It is traversed four times backwards and forwards in four different vessels, by a zigzag horizontal flue or flat pipe, nine inches broad and one deep, ending below in a round pipe, which passes through the bottom of the bath, and receives there into it the top of a small black-lead furnace, the innermost crucible of which contains the fuel. It is surrounded at the distance of an inch by a second crucible, which is enclosed at the same time by the sides of the outermost furnace, the strata of stagnant air between the crucibles serving to prevent the heat being dissipated into the atmosphere by the body of the furnace. A pipe from a double pair of bellows enters the ashpit of the furnace at one side, and supplies a steady but gentle heat to carry on the combustion kindled at first by half an ounce of burning charcoal. So completely is the heat which is disengaged by the burning fuel absorbed by the water in the bath, that the air discharged at the top pipe is generally of the same temperature as the atmosphere. The vessel is made of copper, weighing 2 lbs. per square foot; it is 5½ feet long, 1½ wide, 2 deep, with a bottom 5½ feet long, and 1¾ broad upon an average. Including the zigzag tin-plate flue, and a rim of wrought iron, it weighs altogether 85 lbs. Since the specific heat of copper is to that of water as 94 to 1000, the specific heat of the vessel is equal to that of 8 lbs. of water; for which, therefore, the exact correction is made by leaving 8 lbs. of water out of the 600 or 1000 lbs. used in the experiment.

'In the experiments made with former calorimeters of this kind, the combustion was maintained by a current or draught of a chimney open at bottom, which carried off at the top orifice of the flue a variable quantity of heat, very difficult to estimate.

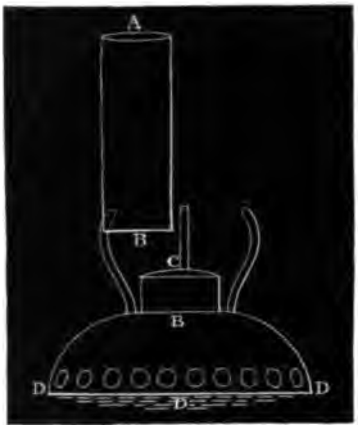
'The heating power of the fuel is measured by the number of degrees of temperature, which the combustion of 1 lb. of it raises 600 or 1000 of water in the bath, the copper substance of the vessel being taken into account. 1 lb. of dry wood charcoal, by its combustion, causes 6000 lbs. of water to become 20° hotter. For the sake of brevity, we shall call this calorific energy 12,000 unities. In like circumstance, 1 lb. of Llangennock coal will yield by combustion 11,500 unities of caloric.'

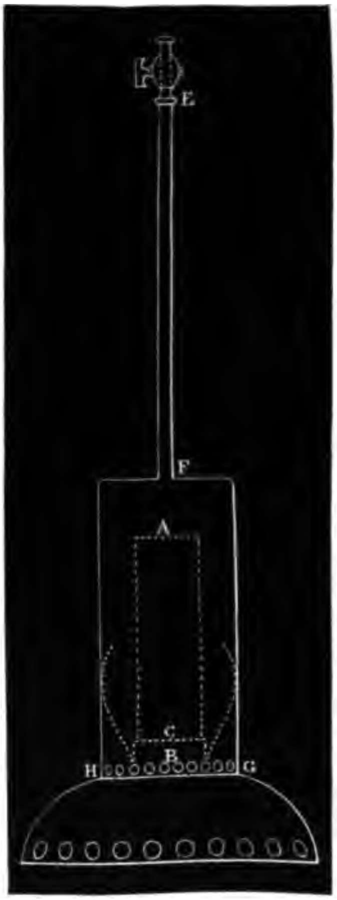
This form of calorimeter of Dr. Ure's seems to possess many advantages over Laplace's and others, and is, no doubt, very convenient in use, although rather bulky.

The instrument known as Wright's calorimeter gives very accurate results, and is the one most generally used now in experiments on the heating power of fuel, in all but the most refined investigations. It is shown in the accompanying figure.

The copper cylinder A B is filled with a mixture of 20 grains of the combustible, and 240 of the deflagrating compound, which is composed of three parts of chlorate of potash, and one of nitrate of potash. A little piece of cotton soaked in chlorate of potash is placed partly in the mixture, the other end projecting above the top of the cylinder; this is ignited, quickly covered with the bell-shaped part of the apparatus, and immersed in a measured quantity of water. As constructed,

FIG. 65.

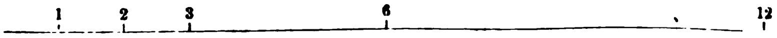




DIMENSIONS.

AB=3¼ in.; diameter ⅝ in.
CD=2 in.
CB, socket for AB.
Diameter across D=4 in.
EF=6 in.
FG=5 in.; diameter HG=1⅝ in.
AB weighs 39½ grammes.
The remainder of the apparatus, including the stop-cock, weighs 391 grammes.

SCALE OF 12 INCHES.



the whole metallic apparatus weighs 6642·7 grains, and with this weight 290·1 grains of water are used. The temperature is recorded before and after making the experiment. During the deflagration the stop-cock is closed, which is, however, opened before taking the temperature the second time. A tenth of the temperature that the water is raised by the combustion, is added for errors that are incidental to the use of the instrument.

If the instrument is made of the weight above given, the result is obtained by a very simple calculation. Each Fahrenheit degree by which the temperature of water has been augmented, corresponds to a pound of water converted into steam.

EXAMPLE.

Fahr.
Temp. of water before making experiment = 56°
„ „ after the combustion . = 65
$9° + \frac{1}{10}$th
„ produced by the combustion = 9·9

20 grains of the coal will convert into steam (maximum effect) 9·9 lbs. of water

$$\frac{9{\cdot}9 \times 7000 \times 2240}{20} = \text{Effect of a ton.}$$

5. **Determination of the Specific Heating Power.**—This represents the heat produced from a certain volume of fuel. It may be ascertained by multiplying the absolute heating power by the specific gravity.

6. **Determination of the Pyrometric Heating Power.**—By pyrometric heating power is meant the degree of temperature which may be obtained by completely burning the fuel. This heating power not only depends upon the composition of the fuel, but chiefly on the time required for its combustion, and this again depends on the looseness and inflammability of the fuel. The absolute heating power of hydrogen is greater than that of carbon, but with regard to the pyrometric heating power it will be found that reverse is the case.

Carbon burned in contact with the air to carbonic acid will produce a heat of 2558° C.; if burned to carbonic oxide it only produces 1310°; hydrogen burning to water will produce a heat of 2080°. From this we learn that fuel rich in carbon, such as anthracite, coal, and coke, will produce a greater pyrometric effect than fuel rich in hydrogen, as wood, &c.

Density is an essential quality of fuel required to produce great pyrometric effect. This is proved in the following way.

When atmospheric air first acts on the carbon contained in fuel, carbonic acid is formed, and the temperature rises to a certain degree, but on passing over glowing coal, carbonic acid becomes converted into carbonic oxide, and this causes a portion of the heat at first produced to become latent. This conversion of carbonic acid into carbonic oxide is more easy and complete, as the fuel used is more inflammable; and as a greater quantity of heat is thereby rendered latent, it follows that the heating power of such a fuel is inferior. This accords with general experience; for it is well known that coke is able to produce a greater heat than charcoal.

Several good methods for determining pyrometric heating power were given in the last chapter.

7. **Determination of the Volatile Products of Carbonisation.**—The amount of volatile matter yield on carbonising a fuel depends partly on the composition of the fuel, and partly on the temperature employed. If a fuel rich in oxygen and hydrogen is quickly heated, it will yield the greatest amount of volatile products. These are partly liquid (tar, naphtha, and acetic or ammoniacal water), and partly gaseous (carbonic oxide, carbonic acid, and light and heavy carburetted hydrogen). The more oxygen a fuel contains, the more carbonic acid and carbonic oxide it will produce; the more hydrogen it contains, the more illuminating gas it yields. The applicability of a sample of coal to the production of illuminating gas depends on these conditions.

Coal distilled at a low temperature yields much tar and comparatively little gas, and when a very high temperature has been used, less tar and more gas is produced, but the great heat will have reacted on the gas and injured its illuminating qualities. If the coal contains pyrites, the gas will contain sulphur compounds. The amount of water produced is generally larger than that of the tar.

In order to estimate the amount of volatile matter given off from any particular sample of coal, proceed in the following manner:—Place a given weight, say 200 grains, of the coal in an iron tube, closed at one end, to the other end of which adapt, by means of a cork, a glass or other tube, which must be conducted into an inverted jar full of water standing in the pneumatic trough. Raise the temperature very gradually to redness, and continue the heat until no more gas is given off, then ascertain its quantity in cubic inches, with due correction for temperature and pressure.

8. **Examination of the Coke or Charcoal left behind on Carbonisation.**—The amount of coke or charcoal yielded by a sample of fuel is found by the last operation. The residue is the amount of coke which that particular sample of coal produces; and its weight, divided by two, gives the percentage of coke.

The process of coking, charring, or carbonising fuel, whilst it drives off some of the valuable hydrocarbon constituents, also gets rid of all the aqueous elements. And, therefore, the coke or charcoal which is left behind has its value greatly increased when high temperatures are required, although, from the absence of flame-yielding constituents, it is much more difficult to ignite.

The degree of inflammability of coke or charcoal is relatively the same as that of the raw fuel from which they were produced. The more inflammable a fuel has been, the more inflammable will be the coke or charcoal produced from it.

The temperature employed in the carbonisation, as has been already explained, exerts great influence on the yield of coke.

If the fuel contains iron pyrites, part of the sulphur goes off in the volatile portion, but from $\frac{1}{4}$ to $\frac{1}{2}$ is retained in the form of FeS.

9. **Determination of the Amount of Ash.**—In order to ascertain the amount of ash:—Fully ignite about 50 grains of coal in a platinum capsule, allowing the air to have free access all the time until nothing but ash is left. Its amount may then be ascertained by weighing: good fuel should contain little ash. It may vary from 1 to 10 per cent.,

but if it exceeds 5 per cent., it becomes deleterious. The chemical composition of the ash also influences the quality of the fuel to some extent.

10. **Determination of the Amount of Sulphur.**—This is an important operation in the assay; as a coal containing sulphur cannot be employed for particular operations, and, indeed, those which contain much sulphur ought only to be used for the commonest purposes. This assay is most important to steamboat and other companies, who consume fuel under steam-boilers; and the coal they purchase should always be subjected to this particular test, as sulphur has a corroding and destroying action on iron and copper. Where sulphurous coals are continually burnt under boilers, the metal of the latter becomes deteriorated, and the boiler is rapidly rendered useless. Sulphur exists in coal in the form of iron pyrites; these can generally be detected by their brassy colour. Some coals and lignites also contain sulphate of lime, and in rare cases sulphate of baryta.

The process for the determination of the amount of sulphur in coal is not difficult. 1 part of the coal is to be finely pulverised, and then mixed with 7 or 8 parts of nitrate of potash, 16 parts of common salt, and 4 parts of carbonate of potash, all of which must be perfectly pure; the mixture is then placed in a platinum crucible and gently heated. At a certain temperature, the whole ignites and burns quietly. The heat is then increased until the mass is fused: the operation is finished when the mass is white. It must, when cold, be dissolved in water, the solution slightly acidulated by means of hydrochloric acid, and chloride of barium added to it as long as a white precipitate forms. This precipitate is sulphate of baryta, which must be collected on a filter, washed, dried, ignited, the filter burnt away, and the remaining sulphate of baryta weighed: every 116 parts of it indicate 16 of sulphur.

Dr. Price has drawn attention to a source of error which has hitherto escaped notice in the estimation of sulphur, where fusion of the substance with nitre is the process employed. This author has found that unless great care be taken to prevent the fused mass passing over to the outside of the vessel, and so coming in contact with the flame, or products of combustion, an appreciable and, in some cases, serious error will arise, owing to the sulphuric acid produced from the sulphurous acid in the flame—a product of the oxidation of the sulphide of carbon contained in the gas —combining with the potash of the fused salt. Several experiments have been made to ascertain the amount of error that may be occasioned from the above cause. In one instance, the flame issuing from a Bunsen's burner was made to strike against a little fused nitre on the underside of a small platinum dish, when, in three quarters of an hour, as much sulphuric acid was obtained as is equivalent to 12 milligrammes of sulphur. As a check on these experiments, nitre was fused by the flame of the spirit lamp, when, as was to be anticipated, not a trace of sulphuric acid could be detected upon the addition of a barium-salt to the aqueous solution of this fused mass, rendered acid by hydrochloric acid. In determinations of sulphur in coke or coal, great care should, therefore, be taken to prevent any of the fused saline contents of the crucible from getting on to the outside. In fusing pig-iron with nitre, a process recommended by some for the estimation of the sulphur it contains, the mass, especially if the iron be rich in manganese, invariably creeps over to the outer wall of the crucible; and it is, therefore, impossible to obtain correct results when the operation is conducted over the gas flame. The analyst should for these reasons always employ a spirit flame in preference to gas in sulphur determinations.

There is another process for the estimation of sulphur which is sometimes adopted, as it is quicker and less troublesome than the above; this is by oxidising the coal by boiling it in nitric acid, instead of fusing it with nitre. Mr. Crossley[*] has, however, shown that this plan gives very incorrect results.

11. **Examination of other Peculiarities of Fuel.**—Besides the above-named examinations, the assayist should

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[*] *Chemical News*, May 1862.

notice the degree of inflammability of the fuel, and whether any particular smell is evolved during combustion; whether the coal is good for coking purposes; whether it burns with a large smoky flame or a luminous flame; whether it burns quietly or with decrepitation; and whether the ash is dusty or fusible, and likely to accumulate and clog up the grate bars.

## CHAPTER VI.

### REDUCING AND OXIDISING AGENTS—FLUXES, ETC.

In some of the operations in the dry way, bodies are heated in suitable vessels *per se*; but it is more often necessary to add to the bodies submitted to assay other substances, which are varied according to the nature of the change to be effected. These substances may be divided into five classes:— I. reducing agents; II. oxidising agents; III. desulphurising agents; IV. sulphurising agents; and lastly, V. fluxes properly so called.

### I. REDUCING AGENTS.

The substances belonging to this class have the power of removing oxygen from those bodies with which it may be combined. In assaying, the substance under examination is generally fully oxidised either naturally or artificially, before reduction is required to be effected. The most common reducing agents are as follows:—

1. Hydrogen gas.
2. Carbon.
3. The fat oils, tallow, pitch, and resins.
4. Sugar, starch, and gum.
5. Tartaric acid.
6. Oxalic acid.
7. Metallic iron, and lead.

**Hydrogen Gas.**—The most common method of preparing this gas consists in dissolving zinc in dilute sulphuric acid. But as this plan gives the gas in the moist state, it must be dried, by being allowed to bubble through oil of vitriol or by being passed through a bottle containing fragments of dried chloride of calcium, before it is used for assaying purposes. This gas will only be required in very accurate assays, which are generally performed where there are ample conveniences for generating pure hydrogen gas. The gas is inodorous, invisible, and colourless, when absolutely pure. It is a most powerful reducing agent, and reduces a great number of metallic oxides at a red or white heat; viz. the oxides of lead, bismuth, copper, antimony, iron, cobalt, nickel, tungsten, molybdenum, and uranium. When any metal is required in a state of absolute purity, this is the only reducing agent admissible, as others give the metal combined with a certain proportion of carbon.

**Carbon.**—Found in large quantities in the mineral kingdom, but generally combined with other bodies. In a perfect state of purity, it constitutes the diamond. The diamond, like all other species of carbon, is unacted on by the highest possible temperature when in close vessels. It burns in atmospheric air and oxygen gas, but requires for combustion a higher temperature than ordinary charcoal. After the diamond, the varieties of carbon found in nature or artificially prepared, are:—

Firstly, **Black-lead** or **Graphite.**—This is a mineral found in beds in the primitive formations, principally in granite and mica-schist. It is generally mixed with earthy substances, and rarely yields less than 10 per cent. of ash. Before employing it for reduction purposes it should be purified. Löwe[*] has given an excellent plan for effecting this object.

Secondly, **Anthracite.**—Which is another species of fossil carbon much resembling ordinary coal, but differing from it by burning with neither smell, smoke, nor flame.

Thirdly, **Coke.**—Which is the residue of the coal employed in the gas-works after all the volatile matter is expelled. It is generally iron black, and has nearly a metallic lustre; it is difficult to inflame, and burns well only in small pieces, but gives a very intense heat. Oven or furnace coke is preferable, as it is harder, lasts longer, and is more economical in use.

Fourthly, **Wood Charcoal.**—Which is obtained by burning the woody part of plants, with a limited supply of air, so

[*] Polyt. Centr. 1855, p. 1404.

as to drive off all their volatile matters, and leave merely their carbon. It is this kind that is generally employed in assays. It ought to be chosen with care, well pulverised, passed through a sieve, and preserved in well-stopped vessels. Wood charcoal is never perfectly pure, generally containing a proportion of hydrogen and watery vapour: these bodies are not generally prejudicial, but in some experiments they ought not to be present: in that case, pure charcoal may be procured by heating sugar to redness in a close crucible.

The advantage of carbon as a reducing agent consists in its great affinity for oxygen, which at a red heat surpasses that of most other substances. Charcoal by itself possesses two inconveniences: firstly, it has the property of combining with many metals; and in the second place, it is infusible, and cannot combine with vitreous substances. The property it possesses of combining with iron, nickel, cobalt, &c., is of no consequence to the assayer, for the increase of weight it gives is not material, excepting under the circumstances to be hereafter pointed out; but its infusibility and inability to combine with fluxes is a very serious inconvenience; for after the reduction, that portion which has not been consumed remains disseminated with the grains of metal in the fused slag, and prevents the separation of all the metal, and the consequent formation of a good button: a large quantity of charcoal can thus irreparably injure an assay. This inconvenience does not happen, however, when an oxide is reduced by cementation in a lined crucible, but there are some cases in which its employment is inadmissible.

Coke should never be used as a reducing agent in assays, when it is possible to avoid it. It often contains a very large proportion of earthy and other extraneous matters (more particularly sulphur, which is very injurious). Coke is never so good as wood charcoal as a reducing agent, because it burns more slowly. When it is used, the temperature employed for an assay must be much increased.

Coal is nearly always inconvenient, because it swells by heat; nevertheless, as it is not required in very large quantity, it is sometimes employed, being finely powdered and sifted previous to use.

**The Fat Oils.**—The name oil is generally given to those bodies that are fat and unctuous to the touch, more or less fluid, insoluble in water, and combustible. They all become solid at various degrees of temperature. There are some which, at the temperature of our climate, have constantly a solid form, as butter, palm-oil, cocoanut-oil, &c.

**Tallow** is an animal product analogous to the fat oils in properties.

**Resins.**—The greater part of the resins are solid, but some are soft. They are brittle, with a vitreous and shining fracture, and are often transparent. They are very fusible, but cannot be raised to their boiling-point without partial decomposition.

Although all the bodies just mentioned consume in their combustion a large quantity of oxygen, they cannot generally effect the total reduction of an oxide on account of their volatility; so that, before the temperature at which the reduction takes place can be attained, the greater part of the reducing agent has been expelled. They generally act only by virtue of the small carbonaceous residue produced by the action of heat; so that their use is very limited and uncertain. Whenever they are employed as reducing agents, without covering the substance, a loss is experienced, on account of the bubbling and boiling caused by their decomposition: this will always take place unless the contents of the crucible be covered with charcoal powder. Oils are very serviceable in the reduction of a large mass of oxide by cementation; in this case, after the oxide has been placed in the crucible, as much oil is added as the oxide and the lining of the crucible will soak up. Fat or resin is also used to prevent the oxidation of the surface of a metallic bath (as in the fusion of bar-lead samples), by coating the metal, and preventing the action of the atmospheric oxygen.

**Sugar** in its decomposition by heat leaves a much larger proportion of carbon than the oils, fats, or resins; so that it would appear serviceable as a reducing agent. There are some cases in which it may be used with advantage, but it undergoes a great increase in volume when heated; so that losses in an assay may occur by the use of this agent. To purify sugar from mineral ingredients, it should be recrystallised from alcohol. It then may be used as such, or after carbonisation. It yields about 14 per cent. of charcoal: this is pure carbon, and leaves no residue when burnt; it is, therefore, preferable to wood charcoal in cases where no foreign matter should be introduced into the assay.

**Starch**, well dried, and, better still, torrefied, is employed with advantage as a reducing agent, and is better than sugar, as it neither fuses, swells up, nor spirts, and in many cases is even preferable to charcoal, because it is in such a fine state of division that it can be more readily and intimately mixed with the substance to be reduced. Wheat and rye flour have nearly the same qualities as starch. They are sometimes used.

**Gum** decrepitates slightly by heat, softens, agglomerates, and boils, without spirting. The gums can be employed as reducing agents under the same circumstances as sugar and starch, but the two latter are preferable, because they contain no earthy substances.

**Tartaric Acid** is the reducing agent in the cream of tartar, or argol $(KO,\bar{T},HO,\bar{T})$, of which so frequent use is made; but the acid is never employed by itself. When heated in close vessels, it fuses and decomposes, giving off combustible gases, leaving a little charcoal. It burns when heated in contact with air, giving rise to a peculiar and not unpleasant odour.

**Oxalic Acid** fuses at a temperature of 208° without decomposing, but when heated to 230° it is decomposed, giving rise to carbonic acid, carbonic oxide, and a little formic acid vapour, and when heated strongly, some portions volatilise without decomposition; it does not leave a carbonaceous residue.

The property which oxalic acid possesses of not leaving a residue would render it remarkably valuable for the reduction of the metallic oxides in cases where the slightest trace of carbon is to be avoided, if its reducing power were greater; but it decomposes at a low temperature, and in burning absorbs but a small quantity of oxygen, especially when it has not been dried; so that even for the most easily reducible oxides a large proportion must be employed. When it is combined with a base, as potash in binoxalate of potash, its reducing power is much augmented.

**Oxalate of Ammonia**, when heated in close vessels, is decomposed. Its reducing power is nearly double that of oxalic acid.

**Comparative Reducing Power of the above Agents.**—In order to give an idea of the comparative reducing power of the agents just described, the result of some assays made on them by Berthier, by means of litharge, are given below.

By heating the same weight of each reducing agent with an excess of litharge, buttons of lead were obtained, whose weights were proportional to the quantity of oxygen absorbed, and by comparing them with each other the reducing power of each flux is given; by taking for unity the weight of the reagent, calculation has proved that 1 part of pure carbon reduces from litharge 34·31 of lead. The following are the results of Berthier's experiments:—

| | |
|---|---|
| Hydrogen | 104·00 |
| Pure carbon | 34·31 |
| Calcined wood charcoal | 31·81 |
| Amber resin | 30·00 |
| Ordinary wood charcoal | 28·00 |
| Animal oil | 17·40 |
| Tallow | 15·20 |
| Resin | 14·50 |
| Sugar | 14·50 |
| Torrefied starch | 13·00 |
| Common starch | 11·50 |
| Gum-arabic | 11·00 |
| Tartaric acid | 6·00 |
| Oxalate of ammonia | 1·70 |
| Oxalic acid | ·90 |

It must be borne in mind that these numbers do not represent the quantities of oxygen each reagent would absorb in complete combustion; but that it only indicates the quantity of metal produced by equal weights of the reagents.

In assaying, however, it is rarely that these agents are used by themselves; they are generally mixed with a flux properly so called, and under the head of Fluxes they will be more particularly described.

**Metallic Iron** removes oxygen from the oxides of lead, bismuth, copper, &c., but is rarely added for that especial purpose; and when it does produce this effect, it is generally secondary, because it previously existed in the matter subjected to assay, or was added for some other purpose.

**Metallic Lead** reduces but a very small number of oxides, but it reduces many to the minimum of oxidation; it also decomposes some sulphates and arseniates.

## II. OXIDISING AGENTS.

The oxidising agents in general use are as follows :—

1. Oxygen, atmospheric, or combined.
2. Litharge and ceruse.
3. Silicates and borates of lead.
4. Nitrate of potash.
5. Nitrate of lead.
6. Peroxide of manganese.
7. Oxide of copper.
8. Peroxide of iron.
9. The caustic alkalies.
10. The alkaline carbonates.
11. The sulphates of lead, copper, and iron.
12. Sulphate of soda.

**Oxygen** is a gas which has neither smell nor taste, and is about one-tenth heavier than atmospheric air. It has the property of forming compounds with nearly every element, and its affinities are very energetic. Atmospheric air consists of four-fifths nitrogen and one-fifth oxygen.

**Litharge** is a fused protoxide of lead, and is generally obtained from the silver-lead works. When melted, it oxidises nearly all the metals, except mercury, silver, gold, palladium, platinum, &c., and generally forms very fusible compounds with the oxides. These two properties cause it to be a very valuable agent in separating silver and gold from all the substances with which they may be mixed.

Litharge is occasionally mixed with a little of the red oxide of lead; the presence of this in large quantities becomes injurious, as it has the property of oxidising silver. Ordinary litharge can be easily freed from this oxide by fusing it and pouring it into a cold ingot mould, then pulverising, and carefully keeping it from contact with air, as it readily absorbs oxygen, and if it be allowed to cool in the atmosphere, it will nearly all be converted into the red oxide.

**CERUSE**, or **WHITE LEAD**, is a carbonate of the protoxide of lead. As it does not contain the slightest traces of red oxide, it may be used where the presence of that substance may be inconvenient; but it is troublesome to use, as it is much less dense than litharge; large vessels must be employed in consequence; besides, it generally contains a small quantity of acetate or subacetate of lead, and sometimes metallic lead separates from it on ignition, which is, in some cases, disastrous to the result of an experiment. When ceruse is employed, a certain quantity must be fused, to ascertain if any metallic lead be produced;[*] and, on the other hand, it must be examined to ascertain if it be adulterated with sulphate of baryta. When it is pure, it dissolves completely in acetic or nitric acid.

**ACTION OF OXIDE OF LEAD ON THE METALS.**—The following are the results of the experiments of Berthier on the action exercised by oxide of lead on sulphur, selenium, tellurium, arsenic, and the most common of the metals. The experiments were made in a furnace capable of producing heat enough for a copper assay.

**SULPHUR.**—Oxide of lead is completely reduced by sulphur, with the formation of sulphurous acid, but not a trace of sulphuric acid: thus $S+2PbO = 2Pb+SO_2$.

**SELENIUM** is dissolved by oxide of lead in all proportions; but these bodies exercise no action on each other.

**TELLURIUM** is strongly attacked and converted into telluric acid, which combines with the oxide of lead when the latter is in excess ($Te+4PbO=3Pb+PbO,TeO_3$). If the contrary be the case, the excess of acid is volatilised and telluride of lead produced ($2Te+3PbO=Te3Pb+TeO_3$).

**ARSENIC.**—When metallic arsenic is heated with litharge, if the latter be employed in great excess, all the arsenic is oxidised ($As+3PbO=AsO_3+3Pb$); if not, a part only is oxidised, and lead reduced; the remainder volatilises, or forms an arsenide of lead. (For nature of reaction, refer to the preceding metal, Tellurium). Mixtures of:

---

[*] Berthier.

| | | | |
|---|---|---|---|
| Arsenic | 75·24 | 37·60 | 9·40 |
| Litharge | 111·60 | 111·60 | 111·60 |

gave: No. 1, a lamellar metallic button, and a compact vitreous slag of a fine orange-colour. The fusion was accompanied by a considerable amount of arsenical smoke.

No. 2 gave a semiductile metallic button, with a lamellar fracture, like galena, but not so blue, and a transparent vitreous orange-coloured slag.

The third yielded a button of lead and a deep olive-green slag, very crystalline, and in large plates. This fusion was not accompanied by smoke. It is probable that arsenious acid is formed in these reactions: the last slag contained about a fifth of its weight of this acid.

Lead reduces, in part, arsenious acid; in the same manner, arsenic partly reduces oxide of lead. A mixture of

| | |
|---|---|
| Arsenious acid | 12·40 |
| Lead | 38·80 |

produced on fusion a very arsenical vapour, and yielded 32 parts of arsenide of lead, which was deep grey, semiductile, and had a granular fracture; a fine orange-yellow vitreous arsenite of lead was also produced.

**Antimony.**—The two following mixtures of antimony and litharge:

| | | |
|---|---|---|
| Antimony | 10 | 10 |
| Litharge | 40 | 80 |

gave (No. 1), 23 parts of lead, and a compact well-fused slag, of a topaz yellow colour, which contained more than one-third of its weight of protoxide of antimony ($Sb + 3PbO = SbO_3 + 3Pb$). The second gave 26 parts of lead, and a very fluid glass, which cooled rapidly, and was opaque, like yellow wax; it contained:

| | |
|---|---|
| Oxide of lead | 52 |
| Protoxide of antimony | 11·86 |

**Tin.**—This metal, cut into small fragments, was heated with the following quantities of litharge:

| | | | |
|---|---|---|---|
| Tin | 10 | 10 | 10 |
| Litharge | 37·5 | 80 | 120 |

The first mixture gave a slaggy substance, of dull grey colour, not well fused, with globules of lead at the lower part.

The second mixture gave 26 parts of lead, and a semi-fused slag, compact and opaque, the colour yellowish-grey. It contained:

| | |
|---|---|
| Oxide of lead | 52 |
| Protoxide of tin | 11·4 |

The third mixture produced 26·3 of lead, and a very fluid slag, which was compact, opaque, and greyish yellow, with a granular fracture ($Sn+PbO=SnO+Pb$). It contained:

| | |
|---|---|
| Oxide of lead | 97·0 |
| Oxide of tin | 11·4 |

**Zinc.**—Ten parts of zinc filings and 100 of litharge were heated together; as soon as the latter softened, action commenced. A slight bubbling and flaming, occasioned by the combustion of a portion of the zinc, took place, and on increasing the heat the mixture fused completely. The result was a button of lead equal to 13 parts; it was pure and ductile, and was covered with a crystalline slag, like litharge, opaque and yellowish, but in small plates. This experiment proves that about one-fifth of the zinc employed is volatilised, whilst the remainder reduces the litharge ($Zn+PbO=ZnO+Pb$). The slag contains:

| | |
|---|---|
| Oxide of lead | 877 |
| Oxide of zinc | 123 |

**Bismuth.**—Twenty parts of bismuth heated with 40 of litharge, gave a ductile metallic button, tin-white, and weighing 24·3 parts, and a crystalline slag, like litharge.

**Iron.**—M. Berthier heated metallic iron with litharge in the following proportions:

| | | |
|---|---|---|
| Iron wire | 10 | 10 |
| Litharge | 100 | 160 |

The first mixture gave 40 of lead, and a pasty, compact, opaque slag, of a deep metallic black-colour, and very magnetic ($Fe + PbO = FeO + Pb$). There was no metallic iron, but some globules of lead were present. The slag contained about:

| | |
|---|---|
| Oxide of lead | 55·9 |
| Oxide of iron | 13·4 |

The second mixture gave a button of lead, weighing 46·6, and a very fluid, compact, opaque slag, with an unequal shining lustre, deep-brown, and very magnetic. The slag contained, nearly:

| | |
|---|---|
| Oxide of lead | 110 |
| Oxide of iron | 13·4 |

**Copper.**—The following are the results obtained with different mixtures:

| | | | | | |
|---|---|---|---|---|---|
| Copper | 15·8 | 15·8 | 15·8 | 15·8 | 15·8 |
| Litharge | 13·9 | 27·9 | 55·8 | 167·4 | 334·8 |

With the first mixture a button was produced, copper-red on the exterior, grey in the interior, weighing 17 parts; and a compact, opaque, deep-red slag. The slag contained:

| | |
|---|---|
| Oxide of lead | 10·3 |
| Suboxide of copper | 2·4 |

and the button:

| | |
|---|---|
| Copper | 13·6 |
| Lead | 3·4 |

The button produced by the second mixture was exteriorly copper-red, and interiorly grey, spotted with red; it weighed 17·8, and the slag was compact, reddish-brown, and opaque. The slag contained:

| | |
|---|---|
| Oxide of lead | 22·3 |
| Suboxide of copper | 3·6 |

and the button:

| | |
|---|---|
| Copper | 12·4 |
| Lead | 5·2 |

The third mixture gave a button similar to the last, weighing 18 parts, and a compact, opaque, reddish-brown slag. It contained:

| | |
|---|---|
| Oxide of lead | 49·8 |
| Suboxide of copper | 3·8 |

The button was composed of:

| | |
|---|---|
| Copper | 12·4 |
| Lead | 5·6 |

With the fourth mixture, a button weighing 25·6 was produced, and a slightly crystalline, reddish-brown slag, which contained:

| | |
|---|---|
| Oxide of lead | 151·28 |
| Suboxide of copper | 10·32 |

and with the fifth, a grey metallic button, weighing 23·6, and a crystalline slag in large plates, like litharge, yellowish-green and reflecting green. The analysis of the button gave:

| | |
|---|---|
| Copper | 3·6 |
| Lead | 20·0 |

and the slag contained:

| | |
|---|---|
| Oxide of lead | 313·28 |
| Suboxide of copper | 13·72 |

**ACTION OF THE OXIDES OF COPPER UPON LEAD.**—The oxide is speedily reduced to the state of suboxide by excess of lead. If the lead be not in excess, it is totally oxidised, reducing a corresponding quantity of the copper to the minimum of oxidation.

The oxide is reduced to the metallic state by lead, but not completely, because a certain quantity is taken up by litharge. The following mixtures have been made the subject of experiment:

| | | | | | |
|---|---|---|---|---|---|
| Metallic lead | 25·9 | 25·9 | 25·9 | 38·8 | 51·8 |
| Suboxide of copper | 19·8 | 14·9 | 9·9 | 9·9 | 9·9 |

All of these gave an imperfect alloy of copper or lead, and a very fusible slag composed of oxide of lead and suboxide of copper. The first produced a very small globule of copper and a very fluid slag, having a much greater tendency to run through the body of a crucible than litharge. Cooled slowly, it was reddish-brown, opaque, and had a finer texture. It was composed of:

| | |
|---|---|
| Oxide of lead | 27·9 |
| Suboxide of copper | 17·8 |

The second mixture produced a button of copper weighing 4·4, and a deep reddish-brown slag, composed of:

| | |
|---|---|
| Oxide of lead | 27·7 |
| Suboxide of copper | 8·7 |

The button gave:

| | |
|---|---|
| Copper | 4·1 |
| Lead | 0·3 |

The third gave a metallic button weighing 8·8, and a deep red, opaque slag, which contained:

| | |
|---|---|
| Oxide of lead | 24·89 |
| Suboxide of copper | 2·11 |

The button contained:

| | |
|---|---|
| Copper | 5·9 |
| Lead | 2·9 |

In the fourth and fifth mixtures, buttons weighing 21·2 and 34·8 were produced, together with slags similar to the preceding, and containing about 8 per cent. of suboxide of copper.

**Silicates and Borates of Lead** behave as litharge, but they oxidise less rapidly.

They may be prepared by fusing together 1 part of silica or boracic acid with 1 part of litharge. The borates are more fusible than the silicates, but their use is attended with an inconvenience; they swell very much in fusing.

**Nitrates of Potash and Soda** fuse at a temperature below redness, without alteration, but when heated more strongly, they give up oxygen. The action of these salts, when fused, is very energetic, because they have a great tendency to decompose, and because they contain a large quantity of oxygen. They are used as oxidising agents in the purification of the noble metals, and for preparing some fluxes. They ought always to be employed in a state of purity.

Saltpetre often contains impurities. On this account a determination of the real amount of nitrate of potash often becomes necessary, not only in cases where saltpetre is to be used for docimetric purposes, but also when used in certain technical operations, viz. the manufacture of gunpowder, enamel, &c.

If saltpetre is very impure, it may be purified by recrystallisation to such a degree, that it will only contain 2 to 3 per cent. foreign substances (chiefly chloride of sodium).

An exact assay of saltpetre is most difficult, and the different modes in use are not quite exact, on account of the chemical properties of the nitric acid, potash, and soda, which substances are generally contained together in saltpetre, and cannot be perfectly estimated by means of reagents. This is chiefly the case with the nitric acid and soda.

Soda is frequently found in saltpetre, as the manufacturers often intentionally mix the raw saltpetre with soda-saltpetre, and it is also often manufactured from a mixture of soda-saltpetre and carbonate of potash.

The following are the different modes of assaying saltpetre.

*a.* **Huss's Mode.**—This is the most simple mode, and used with success in several parts of Germany.

It is based upon the fact, that the saturation of a certain quantity of water with saltpetre depends on the temperature of the water, and also that a solution of impure saltpetre in hot water crystallises on cooling, the sooner, the more free it is from foreign salts. Huss has determined this degree of saturation for a certain quantity of water at different temperatures, viz. from 8—20¼° R., raising by ¼°, and has averaged the results in a table which shows the percentage of pure saltpetre contained in raw saltpetre.

Forty parts of dried saltpetre are dissolved in 100 parts of pure water of 45—50° R. This is done in a tared glass beaker, which is covered with a glass plate, in order to avoid loss of water by evaporation. The glass plate is furnished with a hole for receiving the thermometer. The water is then stirred till all the saltpetre is dissolved. If during solution insoluble substances (partly of organic origin) are separated, the liquid is to be filtered. The thermometer, divided into ¼ degrees, is put into the solution, and the liquid is stirred all the time, so that the temperature of it is throughout alike; for the same reason the beaker is also put upon thick layers of paper which rest upon wood. The moment the saltpetre begins to crystallise is then to be observed, and, at the same time, the degree of temperature which the solution possesses at that moment. The following table will show the amount of saltpetre contained in solution.

| If a solution of common saltpetre begins to crystallise at the following degrees of Reaumur's thermometer | 100 parts of the assayed saltpetre contain the following parts by weight of pure nitrate of potash. | If a solution of common saltpetre begins to crystallise at the following degrees of Reaumur's thermometer | 100 parts of the assayed saltpetre contain the following parts by weight of pure nitrate of potash. |
|---|---|---|---|
| 8° | 55·7 | 14¼° | 75 |
| 8¼ | 56·3 | 14½ | 75·9 |
| 8½ | 57 | 14¾ | 76·8 |
| 8¾ | 57·7 | 15 | 77·7 |
| 9 | 58·4 | 15¼ | 78·6 |
| 9¼ | 59·1 | 15½ | 79·6 |
| 9½ | 59·8 | 15¾ | 80·5 |
| 9¾ | 60·5 | 16 | 81·5 |
| 10 | 61·3 | 16¼ | 82·4 |
| 10¼ | 62 | 16 | 83·4 |
| 10½ | 62·8 | 16¾ | 84·4 |
| 10¾ | 63·5 | 17 | 85·4 |
| 11 | 64·3 | 17¼ | 86·4 |
| 11¼ | 65 | 17½ | 87·4 |
| 11½ | 65·8 | 17¾ | 88·4 |
| 11¾ | 66·6 | 18 | 89·5 |
| 12 | 67·4 | 18¼ | 90·6 |
| 12¼ | 68·2 | 18½ | 91·7 |
| 12½ | 69 | 18¾ | 92·9 |
| 12¾ | 69·8 | 19 | 94 |
| 13 | 70·7 | 19¼ | 95·2 |
| 13¼ | 71·5 | 19½ | 96·4 |
| 13½ | 72·4 | 19¾ | 97·6 |
| 13¾ | 73·2 | 20 | 98·8 |
| 14 | 74·1 | 20¼ | 100 |

In case a solution does not crystallise at the temperature of 8°, it may be considered as proof that the saltpetre is very impure. In order to determine the amount, it is mixed with an equal part of perfectly pure saltpetre. This mixture is dissolved and determined as before, and the pure saltpetre added is then deducted from the result.

For performing this assay, it is necessary to pulverise the saltpetre as finely as possible, in order to dissolve it quickly. It is also necessary not to use too small quantities. 2½ oz. saltpetre, and 6¼ oz. water, are suitable quantities. The temperature of the water must not exceed 50—55°, otherwise the amount of saltpetre will appear too high (owing to water having been evaporated), and the same error will be produced if the solution is not properly stirred. The best thermometers for this purpose are those filled with alcohol, as the latter expands by heat 8 times more than mercury, and thus a more exact observation of the fractions of the degrees is obtained. When using Huss's table, it is necessary that the thermometers employed should exactly correspond with Huss's thermometer; if this is not the case, the difference between the two thermometers is to be ascertained by trials with solution of pure saltpetre.

*b.* **Gay-Lussac's mode** of assaying saltpetre consists in converting the nitrate of potash into carbonate of potash, and in determining its amount volumetrically by means of standard sulphuric acid. 2·639 grs. saltpetre are mixed with 1 gr. of ignited pine-root, and 12 grs. ignited and finely-pulverised chloride of sodium (the latter is added in order to moderate the combustion), and this mixture is heated in a platinum crucible. After cooling, the mass is extracted by water, and either standard solution of sulphuric acid or oxalic acid is added to the solution. The sulphuric acid is prepared by mixing 70 grs. English sulphuric acid with 600 grs. water, and to this mixture so much water is added again that 100 measures of it will saturate 6,487 grs. carbonate of potash. The number of measures used for saturation will then indicate directly the percentage of carbonate of potash.

The following foreign substances in raw saltpetre should be determined.

*Water.*—Twelve to 20 grammes of air-dried, finely-pulverised saltpetre, are heated in a porcelain crucible to 120° C., and the resulting loss is calculated as water.

*Mechanically-mixed Impurities.*—The substance obtained in the former assay is dissolved in hot water, and filtered through a dried and weighed filter. The residue is well washed with hot water, dried on the filter at 120° C. and weighed. On deducting the weight of the filter, there will be left the weight of the mechanically-mixed impurities (alumina, silica, carbonate of lime, peroxide of iron, &c.), which usually amount to 2 to 5 per cent.

*Lime and Magnesia.*—These substances are precipitated as carbonates in the former filtered solution, raised to the boiling-point, by carbonate of soda; the carbonates are then dissolved in hydrochloric acid, and neutralised with ammonia. The lime can be precipitated by oxalic acid, and filtered off; the magnesia which remains in solution may then be precipitated by phosphate of soda.

The amount of lime in East Indian raw saltpetre which has been once crystallised, varies between 0·216 and 0·265 per cent., the amount of magnesia between 0·263 and 0·28 per cent.

*Chlorine.*—Two to 3 grammes of raw saltpetre are dissolved in about 30 grammes pure warm water, in a flask furnished with a tight fitting stopper, and the amount of chlorine is determined by a standard solution of nitrate of silver. The latter is prepared by dissolving 4·793 grammes of nitrate of silver in 1000 burette divisions of water, each division of the burette will then indicate 0·001 gramme of chlorine. The solution, after being warmed and acidulated with nitric acid, s mixed gradually with the solution of silver; after each addition of the latter, it is to be shaken and then allowed to rest.

The amount of chlorine determined by this assay is calculated as being derived from ⅔ chloride of potassium, and ⅓ chloride of sodium, so that 1 part of chlorine corresponds to 1·927 part of metal (1·285 potassium, 0·642 sodium. Experience has proved that East Indian saltpetre contains chloride of potassium and sodium in these proportions.

*Sulphuric Acid.*—Six to 8 grammes of raw saltpetre are dissolved, and from this solution diluted, and heated to the boiling-point, the sulphuric acid is precipitated by means of a standard solution of baryta. By using a solution of 3·26 grammes nitrate of baryta in 2000 parts of the burette, each division of the latter will correspond to 0·0005 gramme sulphuric acid. The amount of sulphuric acid in East Indian raw saltpetre varies between 0·05 and 0·11 per cent.

*Nitrate of Soda.*—This determination is most difficult, and the following modes are recommended.

α. Perfectly pure potash-saltpetre is mixed with different quantities of soda-saltpetre. These mixtures are put under a glass bell jar, together with a quantity of the raw-saltpetre to be assayed. The glass bell must contain also a vessel with water. After a certain time, it is ascertained which of the standard samples corresponds in weight with the raw saltpetre.

The results so obtained are of value only if no other hygroscopic salts (chloride of magnesium, &c.) are present.

β. Longchamps' mode is based upon the decomposition of soda-saltpetres by chloride of potassium, producing chloride of sodium and nitrate of potassium. The saltpetre is mixed with chloride of potassium, and the solution evaporated down. By this operation, chloride of sodium becomes first separated, and afterwards saltpetre. The latter is washed, dried at 150° C., and weighed. Werther has recommended a similar mode.

γ. According to Ragsky, the flame of alcohol takes a yellow tinge when mixed with saltpetre containing soda.

If the saltpetre does not contain certain oxides, such as alumina, lime, &c. (or if, previously present, they have been precipitated), a solution of antimoniate of potash will precipitate the soda contained in saltpetre solution. The precipitate consists of antimoniate of soda, 100 parts of which contain 84·39 antimonious acid, and 15·61 soda.

The presence of soda is also to be ascertained by washing saltpetre with a saturated solution of pure potash-saltpetre. This saturated solution will then contain a proportionally large amount of nitrate of soda. If a small quantity of the solution is made to crystallise upon a watch glass, soda-saltpetre, showing a rhombohedric form, may be detected by means of a microscope, while potash-saltpetre crystallises in prisms, and chlorides of sodium and potassium in cubes arranged in the form of steps.

*Soda-saltpetre* (*Chili- or cubic-saltpetre*), may, in many cases, be substituted for potash-saltpetre.

[It may be here appropriate to give the method of assaying *gunpowder*.

*a*. The *water* is ascertained by drying a suitable quantity of gunpowder, at 100° C. in an air or water-bath. Two trials must give equal weights.

*b*. To determine the *saltpetre*, the same quantity of gunpowder is moistened with water, finely pulverised, put upon a filter, and washed out completely with hot water. The filtered solution is evaporated to dryness in a small tared porcelain vessel, the mass dried at about 200° C., and weighed as saltpetre.

*c*. To ascertain the amount of *sulphur*, a mixture of 3 grs. gunpowder, 3 grs. carbonate of soda, and 15 grs. chloride of sodium (to moderate the combustion), are heated in a crucible. The resulting mass contains sulphates. After cooling, it is dissolved in water, saturated with nitric acid, and precipitated by chloride of barium. The solution is then warmed and allowed to settle again, the pure solution is poured upon a filter, and the precipitate is stirred up with hot water, and allowed to settle down again. This operation is repeated several times, in order to prevent a portion of the precipitate going through the filter. After having washed it completely, the sulphate of baryta is dried upon the filter, then burned and weighed. 100 parts sulphate of baryta contain 13·71 sulphur.

*d*. The amount of *carbon* is represented by the difference. The quality of it may be ascertained if the residue from solution *b* is boiled with a solution of KS (without free KO). The sulphur will be extracted, and the carbon will remain as a residue; it may then be washed and dried.

The quality of gunpowder may be judged roughly from the amount of saltpetre contained in it. To determine that amount quickly, a hydrometer may be used which indicates the percentage of saltpetre, if a certain quantity of powder is lixiviated with a certain quantity of water. The hydrometers used in Austria are arranged for 400 grammes powder, 1 lb. water, and for a temperature of 17·4° C. The results are exact within ½ per cent.

To estimate the strength of the powder, it is necessary to take into consideration its exterior properties, viz. compactness, size and form of grains, &c.]

**Nitrate of Lead** acts in a similar way to the two last mentioned salts. It is prepared by dissolving litharge in nitric acid, and crystallising the solution.

**Peroxide of Manganese** is easily reduced to the state of protoxide by many metals, and is a very powerful oxidising agent; but is rarely employed, because its compounds are very infusible. It is employed occasionally in the purification of gold and silver.

**Oxide of Copper** is not much employed as a flux, but is often contained in substances submitted to assay; it then acts as an oxidising agent. A great number of metals, even silver, reduce it to the minimum of oxidation: and other metals, as iron, for instance, totally reduce it.

**Peroxide of Iron.**—This, like oxide of copper, sometimes acts accidentally as an oxidising agent.

**The Caustic Alkalies, Potash and Soda**, fuse below a red heat, and volatilise sensibly at a higher temperature. Charcoal, at a high temperature, decomposes the water combined with the hydrates of potash and soda, converting them into carbonates, but an excess at a white heat decomposes the carbonate, and potassium or sodium is the product.

**Carbonates of Potash and Soda** are very much employed as agents in the assay by the dry way. They have the power of oxidising many metals, as iron, zinc, and tin, by the action of the carbonic acid they contain; part of it being decomposed, with the formation of carbonic oxide.

**Sulphates of Lead, Copper, and Iron.**—These three salts at a high temperature oxidise the greater number of the metals, even silver, the sulphuric acid giving off oxygen and sulphurous acid. They are used in the assay of gold.

**Sulphate of Soda** is not used by itself as a reagent, but is a product in many operations; it is either formed in the course of an assay, or is contained as an impurity in some of the bodies used.

### III. DESULPHURISING REAGENTS.

1. The oxygen of the atmosphere.
2. Charcoal.
3. Metallic iron.
4. Litharge.
5. The caustic alkalies.
6. The alkaline carbonates.
7. Nitre.
8. Nitrate of lead.
9. Sulphate of lead.

1. **The Oxygen of the Atmosphere** acts as a desulphurising agent in roasting, combining with the sulphur present, forming sulphurous acid ($2FeS_2 + 11O = Fe_2O_3 + 4SO_2$) or sulphuric acid ($CuS + 4O = CuO,SO_3$), sometimes both.

2. **Charcoal** decomposes many sulphides by taking their sulphur to form sulphide of carbon. It acts in this manner with the sulphides of mercury, antimony, and zinc ($2ZnS + C = 2Zn + CS_2$). It is only employed as an auxiliary to the desulphurising power of the alkalies and their carbonates.

3. **Iron** separates sulphur from lead ($PbS + Fe = Pb + FeS$), silver, mercury, bismuth, zinc, antimony, and tin, but only partially decomposes the sulphide of copper. It is generally used in the state of filings, or nails; the latter are preferable, and ought to be kept free from rust. Oxide of iron may be used if it be mixed with the requisite quantity of charcoal to reduce it. Cast iron must not be employed, as it has very little affinity for sulphur.

4. **Litharge** ($PbO$) exercises a very energetic action on sulphides, even at a low temperature. If it be employed in sufficient proportion, the sulphide acted on is wholly decomposed. The sulphur is often disengaged as sulphurous acid,

and the metal remains alloyed with the lead proceeding from the reduction of a portion of the litharge, or combines as oxide with that portion of the litharge which is not reduced. The quantity of litharge requisite for the decomposition of a sulphide is considerable, and varies according to its nature; some sulphides require 34 times their weight. When less than the requisite quantity is used, only a portion of the sulphide is decomposed, and a corresponding quantity only of lead reduced, whilst the remainder of the sulphide forms, with the litharge and the metallic oxide which can be produced, a compound belonging to the class of oxysulphides, which is generally very fusible.

When the sulphides have a very strong base, as an alkali or alkaline earth, no sulphurous acid is given off by the action of litharge, but all the sulphur is converted into sulphuric acid.

Litharge is a very valuable reagent, and its use is nearly exclusively confined to the assay of sulphides containing the noble metals, as these metals are thus obtained as alloys of lead, which are afterwards assayed by cupellation.

The following is an account of the behaviour of this reagent with the ordinary sulphides.

*Sulphide of Manganese* requires at least six times its weight of litharge to produce a fusible compound, and thirty times its weight to desulphurise it completely. The sulphur and metal oxidise simultaneously ($MnS + 3PbO = MnO + SO_2 + 3Pb$), and a protoxide of manganese is formed, which partly peroxidises, taking a brownish tint in contact with the atmosphere. Berthier assayed the four following mixtures:

| | | | | |
|---|---|---|---|---|
| Sulphide of manganese . | 5 | 5 | 5 | 5 |
| Litharge . . . . | 20 | 30 | 100 | 150 |

The first produced an infusible, greyish-black, scoriform mass, in which small plates, having the look of galena, could be discovered. It was composed of the sulphides and oxides of manganese and lead. Much sulphurous acid was given off during the operation.

The second fused to a soft paste, and gave 17·5 of lead, and a compact, vitreous, opaque slag, of a very deep brown colour. The slag contained about half its weight of sulphide of manganese.

The third fused readily, and produced 31·5 of ductile lead, and a transparent, vitreous slag, of a deep hyacinth red.

The fourth produced 33·7 of lead, exceedingly ductile, and the desulphurisation was complete.

*Sulphide of Iron.*—Thirty parts of litharge are sufficient to scorify protosulphide of iron; the metal is converted into the protoxide ($FeS + 3PbO = FeO + SO_2 + 3Pb$).

The four following mixtures:

| | | | | |
|---|---|---|---|---|
| Protosulphide of iron | 10 | 10 | 10 | 10 |
| Litharge | 60 | 125 | 250 | 300 |

gave, the first a pasty, scoriform mass, colour metallic grey, and very magnetic. It was composed of the sulphides and protoxides of iron and lead.

The second gave a very fluid metallic black slag, very magnetic, opaque, and possessing great lustre, and 36 of lead.

The third gave a compact vitreous transparent slag of a fine resin-red, and 67 of lead.

The last yielded a similar slag to the former, but containing no sulphur, and 70 of lead.

Native iron pyrites was treated with the following proportions of litharge:

| | | | | | | |
|---|---|---|---|---|---|---|
| Iron pyrites | 10 | 10 | 10 | 10 | 10 | 10 |
| Litharge | 60 | 125 | 200 | 300 | 400 | 500 |

The mixtures fused very readily with an abundant disengagement of sulphurous acid.

The first produced only a metallic button, divisible into two parts: the lower was the largest, and was a subsulphide of lead; the other looked like the compact galena, but was magnetic; it was composed essentially of the sulphides of iron and lead, but probably contained a small quantity of their oxides.

The second and third gave black vitreous opaque slags which stained the crucibles brown, together with lead, having a granular fracture, and a deep grey colour: the first button weighed 35, and the second 40. Both samples of lead were contaminated with a small quantity of slag, and contained from $\frac{8}{1000}$ths to $\frac{1}{100}$th of sulphur, and a small quantity of iron.

The slags from the three last mixtures were vitreous, transparent, and of a fine resin-red colour: the buttons of lead weighed 45·4, 54·8, and 86 parts. A much larger proportion of litharge does not produce more than 86 of lead, proving that 50 parts of litharge completely effect the desulphurisation of iron pyrites.

*Sulphide of Copper.*—The following mixtures of sulphide of copper and litharge

| | | | | | |
|---|---|---|---|---|---|
| Sulphide of copper . . | 10 | 10 | 10 | 10 | 10 |
| Litharge . . . | 20 | 30 | 50 | 100 | 250 |

fuse very readily, giving off an abundance of sulphurous acid.

The slags formed were compact, vitreous, opaque, or translucid, and more or less bright red. The copper which they contained was at the minimum of oxidation.

The three first mixtures gave metallic buttons, composed of uncombined lead and sulphide of copper.

The fourth gave 28 of lead, with a little adhering sulphide of copper.

The fifth gave 38·5 of pure ductile lead, the exact quantity that ought to be reduced from litharge by the transformation of the above quantity of sulphide of copper into suboxide and sulphurous acid ($2CuS + 5PbO = Cu_2O + 2SO_2 + 5Pb$).

Sulphide of copper does not combine with litharge; this is an exception to the general rule. It requires about twenty-five times its weight of litharge to decompose it completely. When litharge is combined with a certain quantity of protoxide of copper, it has no action on the sulphide of that metal.

The desulphurisation of copper pyrites requires about 30 parts of litharge.

| | | | | |
|---|---|---|---|---|
| Copper pyrites . . . . | 10 | 10 | 10 | 10 |
| Litharge . . . . . | 50 | 100 | 200 | 300 |

were fused together.

In the first assay the fusion was accompanied with much ebullition, and the mass remained pasty: 6 parts of ductile lead were produced, and a matte similar to galena, but deep grey, with small facets, and a brownish-black vitreous slag.

In the second, much ebullition and swelling up took place: 35 of lead, 45 of matte, and a deep brown vitreous slag, were produced.

In the third assay, 49 of lead was the result. It was covered by a thin layer of matte, and a very shining, deep brown, vitreous, translucid slag.

The last mixture fused readily, almost without ebullition, and gave 72 of lead, and a compact shining slag, of a bright grey, and without the least trace of matte; the desulphurisation was complete ($CuS,FeS + 6PbO = CuO + FeO + 2SO_2$).

*Sulphide of Antimony* has a great tendency to combine with litharge, and it must be heated with at least 25 parts to effect its desulphurisation. By mixing these two substances in the following proportion:

| | | | | | |
|---|---|---|---|---|---|
| Sulphuret of antimony . | 10 | 10 | 10 | 10 | 10 |
| Litharge . . . | 38 | 60 | 100 | 140 | 250 |

the three first mixtures afforded very fluid slags, compact, deep black, and slightly metallic, and buttons of ductile lead, weighing 2, 9, and 26 parts. These slags resemble the black litharge produced at the commencement of a cupellation.

The fourth mixture gave a transparent compact slag, vitreous and shining, having a splendid hyacinth-red colour, and 50 parts of lead.

The last produced 57 of lead, proving the desulphurisation to be complete ($SbS_3 + 9PbO = SbO_3 + 3SO_2 + 9Pb$). The antimony, in this case, exists as protoxide in the slag.

M. Fournet has observed that sulphide of antimony has the property of carrying sulphide of copper, and even sulphide of silver, into the compounds formed with litharge. In one of the experiments which he made, a double sulphide, composed of equal parts of sulphide of silver and sulphide of antimony, was fused with three times its weight of litharge, and gave, firstly, a button of lead, mixed with silver; secondly, a matte like galena; and thirdly, a black slag.

This slag was analysed, and found to contain from 8 to 9 per cent. of silver.

It is probable that all the sulphides having a strong tendency to combine with oxide of lead, have, like sulphide of antimony, the property of determining the scorification of a certain quantity of sulphide of silver; like all the sulphides, which in a state of purity, are completely decomposed by oxide of lead.

*Sulphide of Zinc* must be fused with twenty-five times its weight of litharge to be decomposed. The following mixtures were heated together:

| | | | | |
|---|---|---|---|---|
| Blende . . . . | 24·08 | 12·08 | 10 | 10 |
| Litharge . . . . | 55·78 | 83·68 | 100 | 250 |

However strongly the first mixture was heated, it always remained pasty; 29·2 of a greyish-black lead were produced, which contained ·018 of sulphur and ·008 of zinc. The button was covered by a metallic-looking black substance, intermediate between a matte and a slag: it was composed of the sulphides and oxides of zinc and lead.

The second mixture gave 35·5 of lead and a fluid slag, which was compact, opaque, and black.

The third gave 43 of lead, and a deep grey slag.

The last produced 65 of pure lead ($ZnS + 3PbO \ ZnO + SO_2 + 3Pb$), and a vitreous slag, of an olive-colour, and translucid on the edges.

*Sulphide of Lead.*—Galena and litharge, at a heat just sufficient to fuse them, combine and form an oxysulphide; but if the temperature be increased, the two bodies react on each other, and are mutually decomposed ($PbS + 2PbO = 3Pb + SO_2$). If 2789 parts of litharge be employed to 1496 of lead, or 1865 of litharge to 1000 of galena, nothing but pure lead is obtained. If more litharge be employed, a portion is not decomposed, and covers the lead. If less be employed, the galena is not completely decomposed, and the lead is covered by a matte of subsulphide.

But when litharge is combined with a certain proportion of sulphides or metallic oxides, it completely loses its oxidising power on galena, even at a white heat; so that it can be combined with this substance as with the other sulphides, without effecting its total decomposition.

5, 6. **Caustic Alkalies and their Carbonates.**—All the sulphides are decomposed by caustic alkalies, and their carbonates; but in the latter case carbonaceous matter must be present. In the absence of charcoal, there are some sulphides, as of copper, on which they have no action. In these decompositions alkaline sulphides are formed, and combine with and retain a certain quantity of the sulphide submitted to experiment. The proportion of the sulphide which remains in combination with the alkaline sulphides depends on many circumstances. It is always less when a large proportion of alkali or carbonate has been employed; as it is also when a high degree of temperature has been employed; and the presence of charcoal always much diminishes the proportion. When the metal of a sulphide is very volatile, as mercury or zinc, the decomposition may be perfect.

Potash, as it is sold in commerce, always contains foreign substances, viz. silica, peroxide of iron, sulphate, muriate, phosphate and silicate of potash, soda-salts, etc. and also water.

A partial purification of the potash may be effected by dissolving it in boiling water, which will not dissolve some of the above named foreign substances.

The amount of carbonate of potash contained in potash, may be ascertained by standard solution of oxalic acid, or by tartaric acid. Mohr * recommends for this purpose the use of oxalic acid.

Soda also is never free from foreign substances.

The determination of carbonate of soda may be effected also by tartaric acid, or by a salt of oxalic acid.

When employing a standard solution, sulphuric acid is taken, sometimes oxalic acid, while for the determination of potash, a standard solution of oxalic acid is always used.

Carbonate of ammonia is used for decomposing metallic sulphates which are formed during the roasting process of several sulphur minerals. Sulphate of ammonia is then formed, which is volatile when slightly heated.

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* Mohr, Lehrb. d. Titriermethode, 1855.

7. **Nitre, Saltpetre, or Nitrate of Potash** has a very powerful action on the sulphides: in fact, if not modified by the addition of some inert substance, as an alkaline carbonate or sulphate, explosion may take place, and a portion of the contents of the crucible be thrown out. Where an excess of nitre is used, all the sulphur is converted into sulphuric acid, and every metal but gold and silver oxidised. When only the exact quantity of nitre is employed, that is to say, just as much as is sufficient to burn all the sulphur in the sulphide of those metals which are not very oxidisable, as those of copper, silver, and lead, the metal is obtained in a state of purity, and the whole of the sulphur converted into sulphuric acid; but with the sulphides of the very oxidisable metals, the oxygen of the nitre is divided between the sulphur and the metal.

8. **Nitrate of Lead** possesses the combined properties of nitre and litharge. It is not much used.

9. **Sulphate of Lead** is not used as a reagent, but is often formed in the assay of lead ores. It decomposes sulphide of lead by burning the sulphur ($PbO,SO_3+2PbS=3Pb+2SO_2$). It acts on many other sulphides in a similar manner.

### IV. SULPHURISING REAGENTS.

1. Sulphur.
2. Cinnabar, or sulphide of mercury.
3. Galena.
4. Sulphide of antimony.
5. Iron pyrites.
6. The alkaline persulphides.

1. **Sulphur** fuses at 226°, and at 284° is very liquid. It has very powerful affinities, and combines with the greater number of the metals. That kind generally known as flowers of sulphur ought to be employed; and before use, the presence or absence of earthy matters should be ascertained, by exposing it to a dull red heat temperature in a crucible. The sulphur will go off, and the earthy impurities will be left behind.

Sulphur is principally used in the preparation of the alkaline sulphides and in the assay of some of the noble metals.

2. **Cinnabar** is decomposed by many of the metals, and is a better sulphurising agent than sulphur itself, as it is less volatile.

3. **Galena.**—Many metals, as iron, copper, &c., separate sulphur from lead, while some others, as silver, gold, &c., do not; so that if galena be heated with an alloy of various metals, some of which decompose it, and some do not, the former are transformed into sulphides, and the latter combine with the metallic lead which is produced. It is often employed for this purpose. It is a common ore, and readily procured.

The samples employed must contain no sulphide of antimony, and all the matrix must be carefully separated by sifting and washing.

4. **Sulphide of Antimony** yields its sulphur to many of the metals, but it is only used in the separation of gold from some alloys. In this operation the sulphur combines with the alloyed metals, and the antimony with the gold, for which it has much affinity.

5. **Iron Pyrites** is a persulphide which loses half its sulphur at a white heat. It is much employed in metallurgical operations, but not in assaying.

6. **Alkaline Persulphides** can support a tolerably elevated temperature without losing sulphur, but they have a great tendency to do so, and to this is due their sulphurising power. By their means almost every metal can be made to combine with sulphur. When an alkaline persulphide is heated with a metal, or an oxide of a metal mixed with charcoal, a fused compound, a mixture of the sulphide of the metal and an alkaline sulphide, is obtained.

When they are in combination, they are held together by very feeble affinities, and their decomposition is generally effected by the mere action of water, which dissolves the alkaline sulphide and leaves the other perfectly pure. But with gold, molybdenum, tungsten, antimony, &c., the compound is stable and soluble in water; and it is from this fact that the alkaline sulphides are sometimes employed in the assay of auriferous substances.

In order to effect a sulphurisation by means of the alkaline sulphides, it is much better to use equivalent mixtures of sulphur and alkaline carbonates than to prepare them beforehand. To obtain persulphide of potassium, 46 parts of carbonate of potash, and 54 of flowers of sulphur, must be fused together; and for persulphide of sodium, 40 parts of dry carbonate of soda must be heated with 60 parts of sulphur.

When the mixture is fused in a plain crucible, sulphate of potash, or sulphate of soda, is formed, because part of the alkali is reduced to the metallic state by its affinity for the sulphur, giving up its oxygen to a portion of the sulphur, which becomes sulphuric acid; but when charcoal lined crucibles are used, the carbon combines with the oxygen of the alkali, and no sulphate is produced.

## V. FLUXES.

Fluxes are used in the following cases:—

1stly. To cause the fusion of a body, either difficultly fusible, or infusible by itself.

2ndly. To fuse foreign substances mixed with a metal, in order to allow the latter to separate by its difference of specific gravity.

3rdly. To destroy a compound into which an oxide enters, and which prevents the oxide being reduced by charcoal. Silicate of zinc, for instance, yields no metallic zinc with charcoal, unless it be mixed with a flux capable of combining with the silica.

4thly. To prevent the formation of alloys of some metals with others, as, for instance, in the case of a mixture of the oxides of manganese and iron; when a suitable flux is employed, the iron is obtained in a state of purity, whereas if no flux had been added an alloy would have been obtained. Gold and silver can be separated from many other metals by means of a flux.

5thly. To scorify some of the metals contained in the substance to be assayed, and obtain the others alloyed with a metal contained in the flux, as gold or silver with lead.

6thly. A flux may be employed to obtain a single button of metal, which otherwise would be obtained in globules.

Fluxes are divided into non-metallic and metallic; the non-metallic fluxes are—

1. Silica.
2. Lime.
3. Magnesia
4. Alumina.
5. Silicates of lime and alumina.
6. Glass.
7. Borax (biborate of soda).
8. Fluor-spar (fluoride of calcium).
9. Carbonate of potash.
10. Carbonate of soda.
11. Nitre (nitrate of potash).
12. Common salt (chloride of sodium).
13. Black flux and its equivalents.
14. Argol (bitartrate of potash).
15. Salt of sorrel (binoxalate of potash).
16. Soap.

The metallic fluxes are—

17. Litharge (oxide of lead) and ceruse (carbonate of lead).
18. Glass of lead (silicate of lead).
19. Borate of lead.
20. Sulphate of lead.
21. Oxide of copper.
22. Oxides of iron.

1. **Silica** is employed frequently to cause the fusion of some gangues in assays made at an elevated temperature. Silica combines with all the bases, and forms with them bodies termed silicates, which are more or less fusible.

Quartz is the best form of silica to use. For that purpose it must be strongly heated, and then quenched in cold water. It can then be easily pulverised. In case the quartz takes a yellow or reddish colour on ignition, it must be digested with common muriatic acid.

2, 3, 4, 5. **Lime, Magnesia, Alumina, and their Silicates.**—No simple silicate is readily fusible, so that lime, magnesia, or alumina are employed, according to circumstances, to reduce a simple silicate to such a condition that it will readily fuse in an assay furnace. Sometimes, it may be requisite to use all the above-mentioned earths.

Pure lime, when exposed to atmospheric air, attracts carbonic acid and water so quickly that, in practice, pure carbonate of lime is used in the form of chalk, calcareous spar, or marble, if they are pure. Carbonate of lime frequently contains foreign substances, viz., iron, manganese, alumina, silica, and also carbonate of magnesia. A certain quantity of carbonate of magnesia is, in many cases, advantageous, and alumina and silica are not disadvantageous.

Alumina is never used in the pure state. Washed china-clay which, on burning, becomes white, is used instead. Clay generally contains from 20 to about 40 per cent. alumina, and if it is used for the formation of silicates, a quantitative analysis of its components should first be performed.

6. **Glass** is a very useful flux in certain assays, and being a saturated silicate, it will serve by itself either as a slag or merely as a covering. The kind employed must contain no easily reducible metallic oxides, and it must especially be free from arsenious acid and oxide of lead.

The subjoined analyses of glass from *Bodemann Kerl's Probierkunst* will be found useful. (See p. 186.)

7. **Borax** ($NaO,2BO_3 + 10HO$ and $NaO,2BO_3 + 5HO$).—That kind with 10 atoms, or 47·1 per cent. water, effloresces when exposed to atmospheric air, and the other kind with 5 atoms or 30 per cent. water, does not effloresce, and crystallises in octahedrons. This difference is immaterial for assaying purposes, but it is of importance in purchasing borax.

When borax is heated, it loses its water of crystallisation and undergoes an enormous increase of volume; at a higher temperature, it fuses, and forms a transparent glass, which becomes dull on the surface by exposure to air. Only the

ANALYSES OF DIFFERENT KINDS OF GLASS.

| | $SiO_2$ | KO | NaO | CaO | MgO | MnO | $Al_2O_3$ | $Fe_2O_3$ |
|---|---|---|---|---|---|---|---|---|
| 1–2. French green bottle-glass (Dumas and Berthier) | 53·55 | 5·48 | . . | 29·22 | . . | . . | 6·01 | 5·74 |
| | 60·0 | 3·1 | | 22·3 | . . | 1·2 | 8·0 | 4·0 |
| 3–5. „ „ medicine bottle-glass (Berthier) | 62·0 | . . | 16·4 | 15·6 | 2·2 | . . | 2·4 | 0·7 |
| | 71·6 | 10·6 | . . | 10·0 | . . | 0·3 | 3·0 | 1·5 |
| | 69·6 | 8·0 | 3·0 | 13·0 | 0·6 | . . | 3·6 | 1·6 |
| 6–7. English green bottle-glass, decomposible by wine (Warrington) | 59·0 | 1·7 | 10·0 | 19·9 | 0·5 | . . | 1·2 | 7·0 |
| | 49·0 | 2·0 | 7·2 | 24·7 | 2·0 | . . | 4·1 | 10·1 |
| 8–9. Bohemian white hollow wine-glass (8 by Peligot) | 76·0 | 15·0 | . | 8·0 | . . | . . | 1·0 | |
| | 71·7 | 12·7 | 2·5 | 10·3 | . . | 0·2 | 0·4 | 0·3 |
| 10. „ plate (mirror) glass (Peligot) | 67·7 | 21·0 | . . | 9·9 | . . | . . | 1·4 | |
| 11. „ glass goblet, not decomposible by acids (Struve) | 77·25 | 10·73 | 3·13 | 8·71 | . . | . . | trace | trace |
| 12. „ „ (Peligot) | 77·0 | 14·0 | 8·0 | . . | . . | . . | 1·0 | |
| 13–14. „ combustion-tubes for organic analysis (Rowney and Otto). | 73·13 | 11·49 | 3·07 | 10·43 | . . | . . | 0·30 | 0·13 |
| | 74·0 | 18·5 | . . | 7·2 | . . | . . | 0·1 | |
| 15. Glass goblet from Petersburg, of which 8 per cent. alkalies have been extracted by muriatic acid at the boiling-heat (Struve) | 67·85 | 29·98 | 2·06 | 9·41 | . . | . . | 0·88 | |
| 16. French hollow wine-glass | 69·2 | 15·8 | 3·0 | 7·6 | 2·0 | . . | 1·2 | 0·5 |
| 17. French glass for the apparatus of the laboratories in Paris (Salvetat) | 70·5 | 2·1 | 17·20 | 8·7 | 0·2 | . . | 0·5 | 0·3 |
| 18–19. French window-glass | 68·55 | . . | 12·88 | 16·17 | . . | . . | 2·4 | |
| | 69·6 | . . | 15·2 | 13·3 | . . | . . | 1·8 | |
| 20. French mirror-glass (Berthier) | 72·0 | . . | 17·0 | 6·4 | . . | . . | 2·6 | 1·9 |
| 21–23. English mirror-glass | 77·4 | 3·0 | 13·0 | 5·3 | . . | . . | . . | 0·9 |
| | 78·7 | 1·3 | 11·6 | 6·1 | . . | . . | 2·7 | |
| | 77·9 | 1·7 | 12·4 | 4·8 | . . | . . | 3·6 | |
| 24. Venetian mirror-glass | 68·6 | 6·9 | 8·1 | 11·0 | 1·1 | 0·1 | 1·2 | 0·5 |

fused vitrified borax ought to be used in assays. It must be reduced to powder, and kept in well-closed vessels.

As borax may be regarded as containing free boracic acid, it is an excellent and nearly universal flux; it has the property of forming, like boracic acid, fusible compounds with silica and nearly all the bases, and is preferable to that acid because it is much less volatile. It may be used at a high or a low temperature. It is employed in the assay of gold and silver, because it fuses and combines with most metallic oxides, or in obtaining a *regulus*—that is to say, to separate the metals, their arsenides and sulphides, from any stony matter with which they may be mixed; because this salt is neither oxidising nor desulphurising. It is also employed in the assay of iron and tin ores, as in the presence of charcoal it retains but traces of their oxides, and, indeed, much less than generally remains with the silicates.

8. **Fluor-spar (Fluoride of Calcium)** is rarely employed in assays, but in certain cases is an excellent flux, as will be hereafter shown.

9, 10. **Carbonate of Potash** and **Carbonate of Soda.**—It has been already shown that they possess oxidising and desulphurising power: they will now be considered as fluxes.

They are decomposed in the dry way by silica and the silicates, with the separation of carbonic acid. The presence of charcoal much facilitates this decomposition.

They form fusible compounds with many metallic oxides. In these combinations the oxide replaces a certain quantity of carbonic acid; but they are not stable—they are decomposed by carbon, which reduces the oxides, or by water, which dissolves the alkali.

On account of their great fusibility, the alkaline carbonates can retain in suspension, without losing their fluidity, a large proportion of pulverised infusible substances, as an earth, charcoal, &c.

The alkaline carbonates ought to be deprived of their water of crystallisation, for assaying purposes; in fact, it would be better to fuse them before use. They must in all cases be kept in well-stopped vessels.

They may be used indifferently, but carbonate of soda is to be preferred, as it does not deliquesce, and is generally much cheaper.

The alkaline carbonates of commerce always contain sulphates and chlorides. In some cases this causes no inconvenience, but there are many circumstances in which the presence of sulphuric acid would be injurious.

Carbonate of potash can readily be procured free from sulphate and chloride by means of nitre and charcoal, as follows:—Pulverise, roughly, 6 parts of pure nitre, and mix them with 1 part of charcoal; then project the mixture, spoonful by spoonful, into a red-hot iron crucible. The projection of each spoonful is accompanied by a vivid deflagration, and carbonate of potash is found in a fused state at the bottom of the crucible. It must be pulverised, separated from excess of charcoal, and kept in a dry state for use.

Carbonate of soda may be obtained in much the same way, substituting nitrate of soda for nitrate of potash. Either carbonate may also be obtained in a sufficient state of purity by repeatedly crystallising the commercial carbonates.

11. **Nitrate of Potash.**—Its properties have already been pointed out. The presence of silica or of silicates much assists its decomposition.

12. **Common Salt (Chloride of Sodium**, NaCl) is recommended either mixed with flux, or placed above it, for the purpose of preserving the substance beneath from the action of the atmosphere, or to moderate the action of such bodies as cause much ebullition. It is very useful in lead assays, and is much used in the assay of silver by the wet way. It must be previously pounded, and heated to dull redness in a crucible, to prevent its decrepitation.

Common salt, though containing sulphates, chlorides of calcium, and magnesium, is in most cases sufficiently pure for assaying purposes. If intended for copper assays, it must be previously purified from sulphates.

Plattner* has examined the influence of common salt upon different oxides and sulphates. It does not act upon uncombined oxides of lead and zinc. Sulphate of lead, when melted with it at a dull red heat, becomes liquid, and evolves vapour of chloride of lead. By raising the temperature, and by giving more draught of air, the evolution of such vapour is increased. Common salt acts upon sulphate of zinc in the same way. Oxide of antimony and antimonious acid heated with it at a dull red heat evolve vapour of chloride of antimony though not in a great quantity. Sulphate of copper melted with salt at a red heat becomes converted into chloride of copper and sulphate of soda. Chloride of copper becomes vaporised if air is admitted, and it becomes converted into subchloride of copper by raising the temperature a little, chlorine being then evolved.

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* B. u. h. Ztg. 1854, p. 126.

13. **Black Flux, White Flux,** and **Raw Flux.**—*White flux* is produced by deflagrating together equal parts of saltpetre and argol (crude bitartrate of potash); *black flux*, by deflagrating one part of saltpetre with two or three or more parts of argol. Generally one part of saltpetre and two and a half parts of argol are taken. The finely pulverised and intimate mixture for either flux, before it is deflagrated, is called *raw flux.*

After the saltpetre and argol have been finely pulverised and sifted separately, they are intimately rubbed together, and then deflagrated by throwing the mixture little by little into a low-red-hot crucible, which after each addition is lightly covered over. The deflagration may also be conducted, though less advantageously, by filling the crucible about two-thirds full of the raw flux and then touching it with a red-hot coal or iron. It can only be performed in the open air or under a flue with a strong draft, as the tartaric acid evolves various empyreumatic volatile matters in considerable quantity during its decomposition.

With *white flux* the saltpetre suffices to burn all the charcoal produced by the carbonisation of the tartaric acid, and the result is therefore almost pure carbonate of potash, if pure saltpetre and pure argol have been used. If the latter were impure, the resulting neutral carbonate of potash may contain much, perhaps 10 per cent., of carbonate of lime. White flux works like ordinary carbonate of potash, which is therefore almost always preferred to the far more expensive *flux.*

With the *black flux* the quantity of saltpetre is not sufficient to burn all the coal from the argol, and there remain therefore in the black flux, according as two, two and a half, or three parts of argol were taken, about 5, 8, or 12 per cent. of free carbon, which is mixed in the most intimate manner with the resulting neutral carbonate of potash—more intimately indeed than would be possible by any mechanical means. This charcoal does not hinder the fusing of the assay when the flux is used, and effects or promotes the reduction of the metallic oxides.

Fusion and reduction, sometimes also desulphurisation, are the purposes for which black flux is used, and, according to the special character of the assay, a greater or a less proportion of charcoal to the carbonate of potash may be desirable, and this is to determine whether two, two and a half, three, or more parts of argol are to be used to one of saltpetre. As a general rule it may be stated, the more difficultly fusible is the assay, the more potash; and the more metallic oxide is to be reduced, the more charcoal; and the more also of the latter, the more oxygen the oxide contains.

In many cases, instead of black flux, a *mixture* of carbonate of potash and powdered charcoal, in a suitable ratio to each other, suffices, especially if the mixture, before use, is passed through a sieve, or otherwise very intimately mingled. Instead of the powdered charcoal, also, a corresponding (about two to four times as large) quantity of flour, or sugar, or starch may be mixed with the carbonate of potash. Lamp black is, however, the best form of carbon. The three following fluxes are very useful:—

| | | | |
|---|---|---|---|
| Carbonate of soda . . . . . . | 94 | 88 | 816 |
| Charcoal . . . . . . . . | 6 | 12 | 184 |

The second is very nearly equivalent to sodium and carbonic acid, and the third to sodium and carbonic oxide; but it must be observed, that whatever precautions be taken, these mixtures never become so liquid as black flux, because the charcoal tends very much to separate and rise to the surface.

A mixture of 100 parts of pure carbonate of potash and 10 to 15 parts of wheat or rye flour is to be preferred to black flux in case the argol contains gypsum, or the salt-petre, sulphates, which in many cases might work injuriously upon the assay. If this is the case, then, in the presence of a reducing flux, sulphide of sodium is apt to form, which, for example in the copper assay, occasions the slagging of copper.

Cream of tartar, carbonised by a semi-combustion until it is reduced to half its weight, is a very good substitute for black flux; it contains about 10 per cent. of charcoal.

As a perfectly general rule for the use of black flux, and of mixture similar to it, it is to be observed that the crucible should never be more than two-thirds filled, as the assay always intumesces, *i.e.* evolves gaseous matters, when free carbon is present.

14. **Argol, Cream of Tartar,** or **Bitartrate of Potash.**—When bitartrate of potash is heated in a covered crucible, a rapid decomposition takes place, accompanied by a disengagement of inflammable gases: the substance agglomerates, but without fusing or boiling up. The residue is black and friable, and contains 15 per cent. of carbon when produced from rough tartar or argol, and 7 per cent. from cream of tartar.

These reagents produce the same effects as black flux, and possess more reducing power, because they contain more combustible matter: but this is an inconvenience, for the excess prevents their entering into full fusion when the substance to be assayed requires but a small proportion of a reducing agent. They can be used with success in assays requiring much carbonaceous matter.

15. **Salt of Sorrel,** or **Binoxalate of Potash,** when heated, is decomposed. It decrepitates feebly, and during its decomposition is covered with a blue flame; it at first softens, and when fully fused is wholly converted into carbonate. When the oxalate is very pure, the resulting carbonate is perfectly white, and free from charcoal: but very often it is spotted with blackish marks. It has no very great reducing power.

16. **White, or Mottled Soap**, is a compound of soda with a fat acid. When heated in closed vessels it fuses, boiling up considerably, and during its decomposition gives off smoke and combustible gases, and leaves a residue composed of carbonate of soda with about 5 per cent. of charcoal. Of all reducing agents, soap absorbs the greatest quantity of oxygen; and, as the residue of its decomposition by heat affords but little charcoal, it has the property of forming very fluid slags. Nevertheless, it is rarely employed, because certain inconveniences outweigh its advantages. These inconveniences are, its bubbling up, and its extreme lightness. It also requires to be rasped, in order to mix it perfectly with the substances it is to decompose, and it then occupies a very large volume, and requires correspondingly large crucibles. By mixing rasped soap with binoxalate of potash or carbonate of soda, excellent reducing fluxes may be made:—

| | | |
|---|---|---|
| Salt of sorrel | 85 | 3·35 |
| Soap | 15 | |
| Carbonate of soda | 85 | 2·40 |
| Soap | 15 | |

*Reducing Power of the various Fluxes.*—By fusing equal weights of each of the above-mentioned reducing fluxes with an excess of litharge, the following quantities of lead were yielded:—

| | | |
|---|---|---|
| Common black flux, made with two parts of tartar | | 1·40 |
| Ditto, with 2½ of tartar | | 1·90 |
| Ditto, with 3 of tartar | | 3·80 |
| Carbonate of soda | 94 | 1·80 |
| Charcoal | 6 | |
| Carbonate of soda | 88 | 3·60 |
| Charcoal | 12 | |
| Carbonate of soda | 90 | 1·40 |
| Sugar | 10 | |
| Carbonate of soda | 80 | 2·80 |
| Sugar | 20 | |
| Carbonate of soda | 90 | 1·15 |
| Starch | 10 | |
| Carbonate of soda | 80 | 2·30 |
| Starch | 20 | |
| Crude tartar, argol | | 5·60 |
| Cream of tartar | | 4·50 |
| Ditto, ditto, carbonised | | 3·10 |
| Ditto, ditto, calcined | | 2·20 |
| Binoxalate of potash | | ·90 |
| White soda soap | | 16·00 |

All fluxes containing alkaline and carbonaceous substances are reducing and desulphurising, besides acting as fluxes, properly so called. They also produce another effect which it is useful to know, viz. they have the property of introducing a certain quantity of potassium or sodium into the reduced metal. This was first pointed out by M. Vauquelin.* He found that when oxide of antimony, bismuth, or lead, was fused with an excess of tartar, the metals obtained possessed some peculiar characters, which they owed to the presence of potassium.

## METALLIC FLUXES.

17. **Litharge and Ceruse.**—These bodies always act as fluxes, but at the same time often produce an alloy with the metal contained in the ore to be assayed. Ceruse produces the same fluxing effect as litharge. The former is the better flux, and is very useful in a great number of assays.

18. **Glass of Lead** (*Silicate of Lead*).—The silicates of lead are preferable to litharge in the treatment of substances containing no silica, or which contain earths or oxides not capable of forming a compound with oxide of lead, excepting by the aid of silica. It may be made by fusing 1 part of sand with 4 parts of litharge: if required more fusible, a larger proportion of litharge must be added.

19. **Borate of Lead.**—The borates of lead are better fluxes than the silicates when the substance to be assayed contains free earths; but in order to prevent them swelling up much when fused, they must contain an excess of oxide of lead. The borate of lead containing 90·56 of oxide of lead and 9·44 of boracic acid, is very good. Instead of borate of lead, a mixture of fused borax and litharge may be employed; it is equally serviceable.

20. **Sulphate of Lead** is decomposed by all siliceous matters, and by lime, so that when these substances are present litharge is produced, which fluxes them.

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* Annales des Mines.

21. **Oxide of Copper** is rarely used as a flux for oxidised matters, but is sometimes employed in the assays of gold and zinc, to form an alloy with those metals. In this case a reducing flux must be mixed with the oxide. Metallic copper may be used, but is not so useful, as it cannot be so intimately mixed with the assay.

22. **The Oxides of Iron** are good fluxes for silica and the silicates. They are, however, rarely employed for that purpose; they are more often used to introduce metallic iron into an alloy to collect an infusible, or nearly infusible, metal, by alloying it with iron; such as manganese, tungsten, or molybdenum.

# CHAPTER VII.

## THE BLOWPIPE AND ITS USE.

THE BLOWPIPE was formerly only used by jewellers and workers of metal for producing sufficient heat for soldering certain small portions of their work; and it was not till about the year 1733, that Anton Swab applied it to the analysis of mineral substances. Cronstedt used the blowpipe to ascertain the difference between various mineral substances as to fusibility, &c. In 1765, Von Engeström published Cronstedt's System of Mineralogy, and added to it a Treatise on the Blowpipe, in which he pointed out the processes of Cronstedt.

This work attracted the attention of philosophers to this valuable instrument, and its use became more general. Bergman, after Cronstedt, extended the use of the blowpipe beyond the bounds of mineralogy to the inorganic kingdom, and in his hands this instrument became an invaluable agent for the detection of minute portions of many metallic substances. Bergman treated the greater number of the minerals known in his time with the reagents employed by Cronstedt, described their action, and improved many of the instruments necessary for their performance. In these experiments, Bergman, whose health did not permit him to carry out such a laborious work, was assisted in his mineralogical studies by Gahn, who became particularly expert in the use of the blowpipe. The following is a very good example of the utility of this instrument in practised hands: 'Ekeberg asked Gahn his opinion of the then newly discovered mineral, the oxide of tantalum, and Gahn immediately discovered that it contained tin, although it did not amount to more than 1 per cent.'

Berzelius, after Gahn, was particularly famed for his skill with the blowpipe, and for his improvements in the form of apparatus; and it is from his excellent work on this subject that the principal portion of the descriptive part of Blowpipes, Lamps, Tongs, &c., is derived.

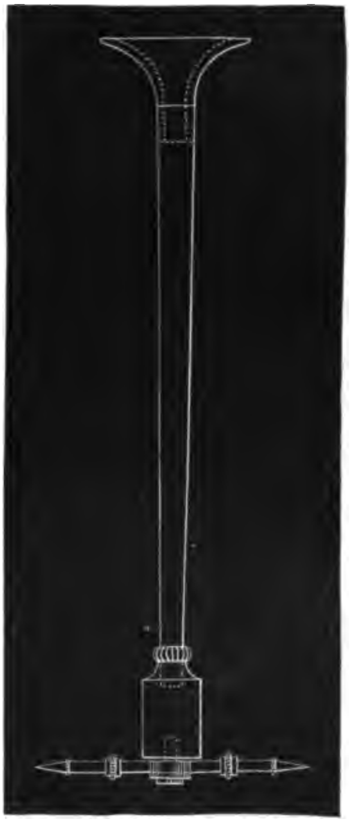
The common blowpipe of gas-fitters, jewellers, &c., is a tube of brass, tapering towards one end, and curved at that extremity, which has an opening as fine as that made by the finest needle; it is this opening which is held against the flame of the lamp, and air is blown to it to increase the amount of heat. In all ordinary operations, the blast is required to be kept up not more than a minute, so that the quantity of moisture exhaled from the lungs produces no inconvenience by stopping up the tube. But in certain chemical operations, this is exceedingly troublesome, as a continuous blast is required, and a large quantity of water collects in consequence, generally sufficient to mar the success of an experiment. In order to obviate this, Cronstedt placed in the centre of his blowpipe a bulb, in which the greater part of the water collected. This form was, however, inconvenient, because if the jet of the blowpipe were at all inclined, even for an instant, the water ran from the bulb, and filled it. In a series of articles communicated to the 'Chemical News,' Mr. David Forbes, F.R.S. has given directions which are invaluable to all who practise with this instrument. From these we quote the following:—

'**Blowpipe.**—The form adopted long ago by Gahn is considered, however, as the most convenient. Fig. 66 shows an improvement made by the author upon this form.

'In this figure it will be seen that the arm of the jet is double, turning upon a central hollow axis, which allows the blast to be directed at will through either half of the arm, merely by rotating the arm itself half round; by having consequently the two holes with respectively a large and small orifice, a corresponding blast may be obtained at pleasure, without suspending the operation.

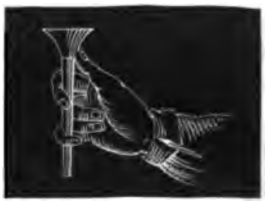
'As a more steady and long-continued blast is required in quantitative operations than could be kept up by using a blowpipe provided with an ordinary mouthpiece held between the lips, without seriously distressing the muscles of the cheeks, it is quite essential that the trumpet mouthpiece, shown in Fig. 66, be adopted; for the same reasons also the mode of holding the blowpipe represented in Fig. 67, is recommended, as securing the greatest steadiness from motion, and as greatly assisting the muscles of the cheeks by the external support afforded them by the position of the thumb pressing against the trumpet mouthpiece.'

FIG. 66.



The nipples are turned, and bored of three different sizes, and are made both of platinum and of brass. The first, of platinum, contains the smallest apertures, and is employed for qualitative analysis; the second, of brass, is used for such qualitative experiments as require a strong oxidising flame, and for heating silver, gold, and copper, in quantitative assay; also for roasting copper, lead, and tin-ores, the metallic contents of which are to be accurately determined; and the third, which is also manufactured of brass, has the largest bore, and is used for the quantitative estimation of lead and tin.

FIG. 67.



Platinum nipples are, however, always preferable to those of brass, because by exposure to a moderate red heat on charcoal before the blowpipe, they are more easily cleaned from the sooty particles which obstruct the aperture. This method of cleansing cannot be applied to brass nipples, owing to their rapid oxidation ; to clean these the operator must adapt to the opening a sharp-pointed fragment of horn, or a small needle, ground along one half of its length ; by this means the aperture through which the air passes may be readily cleaned.

Many persons imagine that the use of the blowpipe is very injurious. Hence, various contrivances have been made to use this instrument by other means ; some have employed double bellows, others bladders, and others, again, the pressure of water ; but none of these methods have afforded satisfactory results, except in the hands of the contrivers, and even in those cases the results have sometimes been very problematical.

Any kind of flame may be used for the blow-pipe, provided it be not too small ; a candle, a lamp, or gas, may be employed : Engeström and Bergman used common candles in preference. Berzelius employs a lamp, which is certainly much preferable to a candle. I have occasionally employed the flame of coal gas, which answers very well, but is not so good as that of a lamp. Berzelius says on this subject, 'Lamps have doubtless many advantages over candles, but are not so convenient in travelling, on account of the escape of oil. The oil employed ought to be the best olive or salad oil.

'The lamp which I use has the advantage of being portable, and closes in such a manner that no oil can escape. It is made of japanned tin-plate, and is about 4 inches long, and 1 inch wide, furnished at one end with a wick-holder, capable of being completely closed by a screw, and at the other with a ring of tin-plate, which passes over the upright end of a support. It may be mentioned, that the screw-cap is furnished with a leather washer, by the aid of which it can be rendered much tighter, and the escape of oil entirely prevented.'

Mr. Forbes says that olive-oil, burnt in the usual Berzelius blowpipe lamp, is probably superior to any other. Gas is not to be recommended, as it is difficult to obtain a good reducing flame when using it. For cupellation and such other operations, however, which only require an oxidating flame, it is excellent.

A spirit-lamp may sometimes be employed in blowpipe assays, particularly when glass tubes are employed, as in the detection of volatile substances. In these cases it is much more convenient; as an oil-lamp, in the first place, blackens the tube; and secondly, does not yield sufficient heat, except when the blowpipe blast is employed.

It is very difficult to give in writing a method whereby a student may acquire the practice of using the blowpipe: that given by the late Professor Faraday * is perhaps the clearest and most concise. He says, 'The practice necessary, in the first place, is that of making the mouth replace the lungs for a short time, by using no other air for the blowpipe than that contained in it.' This practice is simple in itself, and easy to acquire, but, as before stated, difficult to describe. Let the student first observe, that it is easy after having closed the lips to fill the mouth with air, and to retain it so, at the same time that respiration may be carried on; and if, while the mouth is in this state, a blowpipe be introduced between the lips, it will be found that the small quantity of air which its jet allows to pass through it, will be amply supplied for ten or fifteen seconds by the quantity contained in the mouth; and this being repeated a few times, a ready facility for using the blowpipe, independent of the lungs, will be acquired.

This step being taken, the next is to combine this process with the ordinary one of propelling air directly from the lungs through the mouth, in such a way that when the action of the lungs is suspended during inspiration, the blast may be continued by the action of the mouth itself, from the air contained within it. The time of fourteen or fifteen seconds, during which the mouth can supply air independently of the lungs, is far more than that required for one or even many more inspirations; and all that is required to acquire the necessary habit is the power of opening and closing the communication between the mouth and the lungs, and between the air and the lungs, at pleasure.

The capability of closing the passages to the nostrils is very readily proved: every one possesses and uses it when

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* 'Chemical Manipulation.'

he blows from the mouth, and that of closing or opening the mouth to the lungs may be acquired with equal readiness. Applying the blowpipe to the lips as before, use the air in the mouth to produce a current, and when it is about half expended, open the lungs to the mouth, so as to replace the air which has passed through the blowpipe; again cut off the supply, as at first, but continue to send a current through the instrument, and when the second mouthful of air is gone, renew it as before from the lungs.

To some this may be difficult; but if the preceding instructions be followed and persevered in for a short time, the learner will soon find that he can keep up a continuous blast from ten minutes to a quarter of an hour, without any other inconvenience than the mere lassitude of the lips, caused by compressing the mouthpiece of the instrument, and this may be avoided by using the trumpet mouthpiece as recommended by Mr. Forbes.

After having conquered the difficulty of keeping up a continuous blast, the student must learn how to attain the maximum of heat with the least exertion to himself. The chief points to be observed are, neither to blow too fiercely nor too gently; in the first case, the force of the blast would carry away heat by the quantity of cold air thrown into the flame, and in the second, a sufficient amount of heat would not be obtained; because a less amount of air would pass into the flame than that required for perfect combustion.

The highest degree of temperature is required in testing the fusibility of many bodies, as also in the reduction of certain oxides, as those of iron, tin, &c. We have yet another class of phenomena to describe, which do not essentially depend on a high temperature; these are the processes of *reduction* and *oxidation*. In order to explain and point out the best methods of effecting these two objects, it will be necessary to enter somewhat into the nature of flame: this will be done as briefly as is consistent with perspicuity. The species of flame examined will be that of a candle, as it is with a similar one to that with which the blowpipe operator will have to experiment.

On careful examination, it will be found that the flame of a candle or lamp may be divided into four distinct portions: firstly, a deep blue ring at the base; this consists of the vapour of the combustible, which can hardly burn because it has not acquired a sufficient temperature; secondly, a dark cone in the centre; this is also the vapour, but heated intensely, not, however, in a state of combustion, on account of the absence of air; thirdly, of a very brilliant envelope, which surrounds the dark parts just mentioned; this is the partially consumed vapour at a very high temperature; the luminous property it possesses is due to the precipitation and subsequent ignition of particles of solid carbon; and fourthly, of an almost invisible envelope which surrounds the luminous portion; this is the substance of the combustible in full ignition, it here mingles with the atmospheric oxygen, and is consumed. The highest degree of temperature in the whole flame is to be found at the point of contact between the luminous and this part. It must be particularly borne in mind that the inner portions of the flame have an excess of carbonaceous matters, and the outer an excess of oxygenated matters.

Having premised thus much, we will examine the nature of the flame of a candle when acted on by the blowpipe blast, and ascertain how far it is altered, and what are the properties of its separate parts in relation to their oxidising and reducing powers. Supposing the lighted lamp or candle be ready and neatly snuffed, place the nozzle of the blowpipe just in the edge of the flame, and about the sixteenth of an inch above the level of the wick: when things are in this state, blow gently and evenly through the blowpipe, and a conical jet or dart of flame will be produced, which, when formed in a steady atmosphere, free from accidental draughts and currents, will be found to consist of two essential parts—the inner cone, blue, small, and well defined; the outer, brownish and vague. The greatest intensity of heat is found a little beyond the apex of the blue flame; it is there, also, *reduction* takes place. The outer flame is formed by the complete combustion of the combustible matter of the inner; and at that place, and just beyond it, *oxidation* takes place.

*Oxidation*, as before stated, takes place at the extremity of the outer flame, hence it is termed the *oxidising* flame; in it all the combustible portions are super-saturated with oxygen. In general the further the substance to be oxidised can be placed from the extremity of the flame, the better the operation proceeds, provided always that the necessary temperature be maintained. Dull redness is the best suited for oxidation.

*Reduction.*—In this operation the jet of the blowpipe must be introduced into the body of the flame, so as only to produce a small dart; and a jet having a smaller hole than that used for oxidation ought to be employed. By operating thus, a more brilliant flame than the last is produced; it is the result of a less perfect combustion, and therefore contains a large amount of carbonaceous matter, fitting it more especially for the purpose of separating oxygen from all metallic bodies.

Berzelius says, 'the most important point in blowpipe assays is the power of producing *oxidation* and *reduction* at will.' Oxidation is so easy, that to do it requires only to read a description of it; but reduction requires some practice, and a certain knowledge of producing various kinds of blasts. One of the best methods of exercise in this operation is to take a small grain of tin, and place it on charcoal; then direct the blowpipe dart upon it—it will soon fuse; and if the operator has not produced a good reducing flame, it will become covered with a crust of oxide; so that it becomes a witness against him each time this happens. The nature of the flame must be altered until, by observation, the proper kind is produced at will. The longer the button of tin is kept bright, the better and more expert the operator.

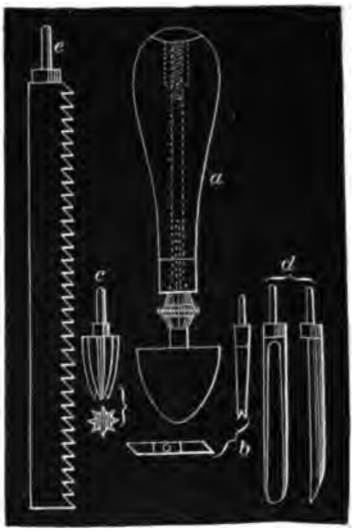
## AUXILIARY BLOWPIPE APPARATUS, ETC.

*Supports.*—The support is the substance destined to hold the material to be assayed whilst under the influence of heat. From this it will be seen that a solid body must necessarily be employed: it ought also to be exceedingly refractory, so as not to give way under the excessive heat; and lastly (with the exception of charcoal), ought to have no chemical action on the substances placed in contact with it. Supports may be divided into combustible and incombustible; the former is charcoal, and for the latter, metal, glass, and earthenware, and in some cases certain minerals have been employed.

*Charcoal.*—Mr. Forbes gives the following excellent description of the preparation of charcoal for blowpipe purposes. 'It is extremely difficult to obtain, in England, charcoal fit for blowpipe operations, without special preparation. The charcoal sold is generally of hard wood, badly burnt, full of cracks, and decrepitating upon application of heat. Good charcoal should be soft, yet compact, and without cracks, and is best made from fir or pine. Where good charcoal cannot be obtained it can be made artificially by moulding charcoal powder agglutinated by some starch paste, and, after desiccation, burning the pieces in a crucible filled with sand.

'For the preparation of the charcoal used as a support for the assays, the instruments represented in fig. 68 are required, all of which are fitted in the universal handle *a*, which is shown in this figure as holding the largest charcoal borer, a section and plan of which are shown in *b*. This large borer is employed for forming the deep holes in the charcoal used in the blowpipe furnace, and which serve to contain the clay crucibles or capsules in which the assays are fused. The blast holes in the charcoal inside the blowpipe furnace are bored out by the gouge-shaped borer *d*, which also serves for making small holes or grooves in charcoal for general purposes. The smaller borer *c*, is most useful, particularly in boring out the holes for receiving the soda paper cornets containing the assay for reduction. The saw-knife *e* also fits into the same handle,

FIG. 68.



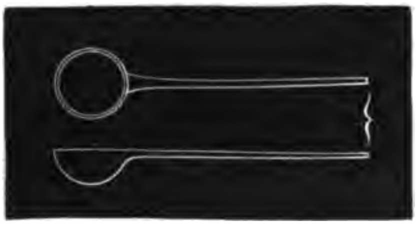
and is used for trimming and sawing across the charcoal pieces, having coarse saw teeth in front, whilst the back presents a sharp knife edge. The figures are all drawn to one-half of the real size.'

*Platinum.*—This metal is much employed as a support in cases where charcoal would be injurious by its reducing power. It is used in three forms, viz. wire, foil, and as a spoon, or small capsule.

*Wire.*—A moderately strong wire of platinum, about 2 inches long, and curved at one end, is used with great advantage in many quantitative examinations. The curve serves as a support in all experiments on tests of oxidation and reduction, where alteration of colour only is to be observed. This support can be relied on, for it is totally free from the false varieties of colour which are too often perceptible when the assay rests on charcoal. In the treatment of metals, or in reduction tests, where an easily melted body is to be operated upon, charcoal must, however, be used. It is necessary to have at hand several platinum wires, so as to proceed to another experiment without being obliged to forcibly remove the adhering borax glass, or to wait for its solution in hydrochloric acid, which is the better mode. If the platinum loop melts with the reagent, it must be cut away, and a new one formed. A wire can be used for a very long time, and when it becomes too short to be held between the fingers the straight end may be fastened into a cork, or a piece of glass tubing.

The platinum spoon (see fig. 69) and foil are used in much the same way; but as charcoal and the platinum wire answer every purpose, it will be unnecessary to describe their use further: small iron spoons of the above form are also made, and are very useful in cases where the presence of iron is not objectionable.

FIG. 69.



Other instruments, as forges, hammer, anvil, agate mortar, scissors, &c., are sufficiently familiar to everybody not to require description. Special apparatus required for any operation will be described in the course of the processes.

**Fluxes and Reagents.**—These most important bodies may be classified under two heads: reagents in the humid way and reagents in the dry way. For the sake of clearness and simplicity, these two classes are subdivided again.

## I. REAGENTS IN THE HUMID WAY.

### *a. Reagents used as Simple Solvents.*

1. *Distilled Water.*—Principally used as a simple solvent for a great variety of substances. It also effects the transformation of several neutral metallic salts (antimony, bismuth) into soluble acid and insoluble basic compounds.

2. *Alcohol.*—It is frequently employed for separating the substances soluble from those insoluble in it (chlorides of barium and strontium); for precipitating bases from their aqueous solutions; for detecting various substances, especially boracic acid, strontia, soda, &c.

3. *Ether* is principally employed for dissolving bromine.

### *b. Reagents principally employed as Chemical Solvents.*

1. *Hydrochloric Acid* is extensively used as a solvent, and for the detection of silver, suboxide of mercury, lead, and ammonia.

2. *Nitric Acid* is employed in the solution of various metals, alloys, and ores, and for the discrimination of certain precipitates. Also as an oxidising agent.

3. *Nitro-hydrochloric Acid.*—Principally used as a solvent for gold and platinum.

4. *Acetic Acid.*—This acid possesses a greater solvent power for certain substances than others, and is accordingly used to separate the former from the latter; as, for distinguishing oxalate of lime from phosphate of lime; the latter being easily dissolved, while the former remains unaltered.

5. *Chloride of Ammonium.*—Used for retaining certain salts and oxides in solution, when others are precipitated by ammonia or some such reagent as magnesia, which is

held in solution by ammonia and chloride of ammonium, when baryta, strontia, and lime are precipitated by carbonate of ammonia.

### c. *Reagents used to separate, or otherwise characterise, Groups of Substances.*

1. *Reagent papers.*

*a. Blue Litmus Paper*, for detecting free acid in solution, its colour being changed to red.

*b. Reddened Litmus Paper*, for the detection of free alkali, its colour being restored to blue.

*c. Brazil-wood Paper*, for detecting hydrofluoric acid, being tinged straw-yellow when immersed in a very dilute solution of this acid.

*d. Turmeric Paper*, for detecting free alkalies; the change produced is very characteristic, its bright yellow colour becoming dark brown.

2. *Sulphuric Acid* has a greater affinity for most bases than any other acid, and is, consequently, employed for liberating them. It is a special test for the detection of baryta, strontia, lead, lime, &c.

3. *Sulphuretted Hydrogen* is an invaluable reagent for separating metals into the principal groups.

4. *Sulphide of Ammonium* divides those metals which are precipitated by sulphuretted hydrogen into two groups; it is also the precipitant of the third group.

5. *Solution of Potash.*—As a precipitant for many oxides it is invaluable. Is used in the separation of iron from alumina, as also nickel from cobalt.

6. *Ammonia* is much employed in the precipitation of metallic oxides, neutralisation of acids, &c.

7. *Carbonate of Ammonia* is employed as a test for many of the earths; it is also used in the removal of an excess of acid from a solution.

8. *Chloride of Barium.*—A special reagent for sulphuric acid. It is also a valuable reagent for subdividing the precipitable acids.

9. *Nitrate of Baryta* acts the same as chloride of barium,

and is substituted for that reagent where the presence of a metallic chloride would be deleterious.

10. *Chloride of Calcium* precipitates, as a group, from neutral solutions, phosphoric, hydrofluoric, oxalic, tartaric, and citric acids; also, if the solution is not very dilute, sulphuric acid.

11. *Nitrate of Silver.*—This is a very important reagent for the classification of acids into groups, and is employed for the detection of certain individual acids, especially hydrochloric, phosphoric, and arsenic acids.

12. *Sesquichloride of iron*, as the reagent for a group of organic acids. It is also employed for the detection of hydroferrocyanic acid in the formation of Prussian blue.

### *d. Reagents used for the Detection of Bases.*

1. *Sulphate of Potash* precipitates from solutions of salts of baryta and strontia the insoluble sulphates of these bases. It also produces a precipitate in concentrated solutions of lime, but only after some time.

2. *Chromate of Potash* precipitates from solutions of the salts of many metallic oxides, chromates; most of them are very difficultly soluble, and possess characteristic colours, by which the particular metal may often be with certainty detected. It is used, principally, however, as a test for lead, with which it gives a yellow precipitate.

3. *Cyanide of Potassium.*—This is a most useful flux. MM. Haidlen and Fresenius say: 'We have examined its action on many oxides, sulphurets, salts, &c., in reference to its use as a reagent combined with the blowpipe. We prefer, in general, a mixture of equal parts of anhydrous soda and cyanide of potassium. This mixture was employed on account of the great facility with which the pure cyanide fuses. It acts, in general, so very similarly to pure soda, that it would be superfluous to describe singly the changes which each individual body appeared to undergo when exposed to its action. We cannot, however, pass over the following especial advantages which it possesses as compared with soda. Firstly, reductions are obtained with such

great facility that the least practised operator may execute reductions which would otherwise be very difficult; for instance, the reduction of tin from either its oxide or sulphuret; and, secondly, that the fused mixture of cyanide of potassium with soda is so easily absorbed by the charcoal, that the grains of reduced metal can always be most distinctly perceived, and may be most easily separated therefrom for further examination.'

Pure cyanide of potassium may be made by heating dry ferrocyanide of potassium to whiteness in close iron vessels, and dissolving the cyanide in alcohol of 60 per cent.; but the salt in its pure state is not used as a blowpipe reagent; it is the mixture of cyanide of potassium with cyanate of potash, formed in the readiest manner by Liebig's process, which may be thus conducted. Eight parts of ferrocyanide of potassium are rendered anhydrous by a gentle heat, and intimately mixed with three parts of dry carbonate of potash; this mixture is thrown into a red-hot earthen crucible, and kept in fusion, with occasional stirring, until gas ceases to be evolved and the resulting mass becomes colourless. The crucible is left at rest for a moment, and the clear salt decanted from the heavy black sediment at the bottom. The ferrocyanide and the carbonate employed ought to be perfectly free from sulphuric acid.

4. *Ferrocyanide of Potassium.*—Principally used for the detection of oxide of copper, and for indicating the presence of sesquioxide of iron.

5. *Ferrocyanide of Potassium* is used as a test for protoxide of iron and its salts.

6. *Sulphocyanide of Potassium*, used like ferrocyanide of potassium, for the detection of sesquioxide of iron.

7. *Phosphate of Soda.*—It precipitates all the alkaline earths, but is chiefly employed, after the separation of baryta, strontia and lime, for the detection of magnesia.

8. *Oxalate of Ammonia.*—This is the most usual test for lime.

9. *Protochloride of Tin* is a very powerful reducing agent, and is employed as a test for mercury and also for gold.

10. *Bichloride of Platinum* is used for the detection of ammonia and potash.

11. *Perchloride of Gold* is a test for the protosalts of tin.

12. *Zinc* is principally employed for the reduction of antimony and tin.

13. *Copper* is used for the reduction of mercurial salts and for the detection of arsenious acid. It also indicates the presence of nitric acid.

14. *Iron wire* is employed to precipitate many metals, and in the separation of sulphur and the fixed acids from any substance with which they may be combined. The metals which can thus be precipitated, or deprived of sulphur, are copper, lead, nickel, and antimony. For instance, if a small piece of iron (harpsichord) wire be placed in a substance in fusion, and acted upon by the blowpipe, it becomes covered with the reduced metal. The latter sometimes appears as small globules.

Iron has the property of reducing phosphorus from phosphoric acid or the phosphates, giving rise to a phosphuret of iron, which forms on fusion a white, brittle, metallic globule.

### *e. Special Reagents employed for determining the Presence of Acids.*

1. *Acetate of Potash* is used for precipitating phosphate of sesquioxide of iron from hydrochloric acid solutions of phosphates of the alkaline earths, and for precipitating from simple solutions in mineral acids substances insoluble in acetic acid.

2. *Hydrate of Lime, Lime Water.* The former, hydrate of lime, is employed for the liberation of ammonia, and the latter for the detection of carbonic acid, oxalic acid, and tartaric acid, and lastly for indicating the presence of citric acid.

3. *Sulphate of Lime*, for distinguishing and separating baryta and strontia.

4. *Sulphate of Magnesia*, for the detection of phosphoric acid.

5. *Chloride of Magnesium* is used for the same purposes as sulphate of magnesia.

6. *Sulphate of Iron.*—Used for indicating the presence of nitric acid; and is also employed in solution for detecting hydroferrocyanic acid and for reducing the salts of gold.

7. *Neutral Acetate of Lead.*—This is specially applied as a test for chromic acid and soluble chromates.

8. *Sulphate of Copper* is used as a test for arsenious acid and arsenic acid. Also, for indicating the presence of hydroferrocyanic acid.

9. *Subnitrate of Mercury.*—This reagent acts analogous to, and in many cases may be substituted for, nitrate of silver, in the detection of chlorine.

10. *Oxide of Mercury* is used as a test for hydrocyanic acid.

11. *Chloride of Mercury* is used as a test for hydriodic acid.

12. *Sulphurous Acid* is a very powerful reducing agent. It precipitates metallic mercury from solutions of mercurial salts, reduces chromic acid to oxide of chromium, and is employed for the conversion of arsenic into arsenious acid.

13. *Chlorine* is used in solution for isolating iodine and bromine.

14. *Sulphindigotic Acid* is used as a test for nitric acid.

15. *Starch Paste* is used as a test for iodine and bromine.

### II. REAGENTS IN THE DRY WAY.

1. **Carbonate of Soda.**—The plain carbonate or the bicarbonate may be indifferently employed; but in either case it is absolutely necessary that it be free from sulphates.

There are two objects in view in the employment of soda as an auxiliary to the blowpipe; firstly, to ascertain if the substances combining with this body be fusible or infusible; and secondly, to facilitate the reduction of certain metallic oxides.

*The Fusion of Substances with Soda.*—Berzelius says, that 'relatively to the employment of soda, there are many things to observe. The necessary quantity must be taken from its receptacle on the moistened point of a knife, and kneaded in the palm of the hand, so that it may form a coherent mass. If the body under examination be pulverulent it must be incorporated with it, but if in lump it must be placed upon it, forcing it slightly into the moistened soda; then carefully heated on the charcoal with a gentle flame, until thoroughly dry; and lastly, it may be fused. It generally happens that the soda, at the instant of fusion, is absorbed by the charcoal; but this does not hinder its action on the assay; for if it be fusible with soda, the latter comes to the surface and attacks it, finally forming a liquid globule. If the substance be infusible in soda, but decomposible by it, it alters its appearance without entering into fusion. But, however, before pronouncing any substance to be infusible by soda, the flux ought to be mixed with the pulverised substance. If in these trials too little soda be taken, a portion of the substance remains solid, and the rest forms a covering of transparent glass; if too much, the bead of glass becomes opaque on cooling. It sometimes happens that the assay contains a substance which, being insoluble in the glass of soda, prevents it becoming transparent. Then, in order that we may fall into no error respecting the nature of the glass, it becomes necessary in the two last-mentioned cases to add a new quantity of the body under examination, and then ascertain if a limpid globule cannot be obtained. In general it is the best method to add the soda by successive small doses, and note the changes produced by each addition. It sometimes happens, in this kind of assay, that the glass becomes coloured at the moment of cooling, and finally takes a yellow or deep hyacinth red; it even becomes occasionally opaque and yellowish brown. These phenomena indicate the presence of sulphur, either in the assay or the soda employed. If the same colour be constantly produced by the same soda, it is a proof that it contains sulphate of soda; it must then be discarded; but if it gives generally a colourless glass, it is the substance under assay that contains sulphur or sulphuric acid.'

*Reduction of Metallic Oxides.*—This species of assay, by which quantities of reducible metals, so small as to escape the best humid analyses, can be detected, is the most important discovery Gahn made in the application of the blowpipe.

If a small quantity of native or artificial oxide of tin be placed on charcoal, it requires a long blast and a skilful operator to produce a grain of metallic tin; but if a small quantity of soda be added, the reduction takes place readily, and so completely with pure oxide, that the whole is transformed into a button of tin. From this it is certain, that the presence of soda favours the decomposition; but in what manner? Berzelius says that the reason is not known.

The action, however, can be explained thus, as Berzelius himself hints: 'The red-hot charcoal reacts upon the carbonate of soda, producing by its reduction a certain amount of sodium, which by its strong attraction for oxygen seizes on that contained by the metallic oxide which is required to be reduced.' If the metallic oxide contain an irreducible substance, the reduction of the former becomes difficult; but if a little borax be added, the reduction takes place as usual.

This assay is very easy of execution, and the metal is moreover readily recognised, as by previous assays the nature of it is somewhat ascertained, and the reduction but confirms the previous idea.

Supposing, however, that the metallic oxide be mixed with such a quantity of non-reducible substances that its nature cannot be ascertained by previous experiment, how can it be proved that a reducible metal is present?

Gahn has solved this question in a very simple manner. 'After having pulverised the substance to be assayed, it is kneaded in the palm of the hand with moistened soda, and the mixture placed on charcoal and exposed to a good reducing flame; a little more soda is then added, and the blast recommenced. As long as any portion of the substance remains on the charcoal, soda is added in small portions, and the blast continued until the charcoal has absorbed the whole of the mass. The first quantities of soda serve to collect the metallic particles scattered in the substance to be assayed, and the final absorption of the latter completes the reduction of any that may remain in the state of oxide.

'This done, the burning charcoal is extinguished with a few drops of water; then having cut out the part which absorbed the soda and assay, grind it to a very fine powder in an agate mortar. This powder is then washed with water to carry away the finest portion of the charcoal. The grinding and washing are repeated until all the charcoal is washed away. If the substance contained no metallic body, nothing will remain in the mortar after this last washing. But if it contained the smallest quantity of reducible matter, it is found at the bottom of the mortar, as small brilliant plates if it be malleable, or as a fine powder if it be brittle or not fusible. In either case, the bottom of the mortar is covered by metallic traces, resulting from the friction of the particles of metal against its sides, (provided that the quantity of metal contained in the sample be not too small). The flattening of almost imperceptible globules of any malleable metal converts them into shining discs of a perceptible diameter. In this manner may be discovered by the blowpipe, in an assay of ordinary size, less than a half per cent. of tin, and even less than that of copper.'

The following points in this class of assay ought to be particularly attended to. Firstly, to produce the strongest possible flame, taking care that it covers every part of the assay. Secondly, to leave none of the metal in the charcoal, or lose the smallest quantity in the collection. Thirdly, to well grind the carbonaceous mass. Fourthly, to decant very slowly, so that only the lighter parts may be carried away by the water. Fifthly, not to judge of the result until the whole of the charcoal has been removed, for a small quantity remaining suffices to hide the metallic particles; and, moreover, the particles of charcoal, viewed in a certain light, have themselves a metallic lustre, which will deceive an inexperienced eye. Sixthly and lastly, not to trust to the naked eye, however plain the sample may be, but always examine by the aid of a good microscope.

The metals reducible by this process are (besides the noble metals), molybdenum, tungsten, antimony, tellurium, bismuth, tin, lead, copper, nickel, cobalt, and iron. Amongst these, antimony, bismuth, and tellurium volatilise easily when they are exposed to a strong heat. Selenium, arsenic, cadmium, zinc, and mercury volatilise so completely that they cannot be collected except by means of a small subliming apparatus.

The reduction can always be effected the first time when the assay contains from 8 to 10 per cent. of metal; but in proportion as the standard decreases more attention and care must be paid to the washing and recognition of the reduced metal in the mortar. A good system of practice in this experiment is to employ any cupreous substance, and make on it a great number of experiments, taking care to mix it each time with a substance containing no copper; thus the metallic value will diminish at each new assay, until at last no copper can be found.

If the substance to be assayed contains several metals, the reduction of their oxides must be made *in globo*, and a metallic alloy obtained. Some, small in number, are reduced separately. For instance, copper and iron give a regulus of each metal; copper and zinc, the first gives a regulus of copper, whilst the latter volatilises. But when the result of the operation is an alloy, recourse must be had to the reactions produced by other fluxes to ascertain its constituents.

2. **Borax (Biborate of Soda).**—The borax of commerce must be dissolved in hot water and recrystallised, before it can be used in blowpipe analysis. Gahn made many experiments on the fusion of borax on charcoal with soda, until both salts were absorbed; a whitish metal was produced, which appeared to proceed from the vessels in which the borax was manufactured. This never happened with borax which had been recrystallised.

Borax may be employed either in crystals, the requisite size for an assay, or in a pulverulent form; in this case it may be taken up on the moistened point of a knife. Some operators prefer fusing the borax before use, in order to drive off its water of crystallisation, and thus avoid the tumefaction ensuing after the heating of a crystal on charcoal. This, as Berzelius observes, would be an excellent precaution, provided the borax did not regain its water of crystallisation, but it recovers it to a small extent, and boils up when exposed to the blowpipe flame, although not so much as before; the tumefaction is, however, no great inconvenience, and it is not difficult to fuse a mass so tumefied into a globule.

Borax is employed in the solution or fusion of a variety of substances. It is best to commence by acting upon a scale of the substance to be examined; because if a powder be employed the resulting action cannot be so well ascertained. The following phenomena are to be carefully watched, for in treating any substance with borax it must be particularly noted whether the fusion takes place rapidly or otherwise; without motion or with effervescence; if the glass resulting from the fusion is coloured, and if that colour changes in the oxidising or reducing flame; and lastly, if the colour diminishes or increases on cooling, and if, under the same circumstances, it loses or retains its transparency.

Some substances possess the property of forming a limpid glass with borax, which preserves its transparency on cooling, but which, if slightly heated in the exterior (oxidising) flame, becomes opaque and milk-white, or coloured when the flame strikes it in an unequal or intermittent manner. The alkaline earths, as yttria, glucina, zirconia; the oxides of cerium, tantalum, titanium, &c., belong to this class. In order to be certain of this result we must assure ourselves that the glass is saturated to a certain point with either of the above class of bodies. The same thing, however, does not happen with silica, alumina, the oxides of iron, manganese, &c., and the presence of silica prevents the production of this phenomenon with the earths; so that alone they present this peculiar appearance with borax; but when combined with silica (as natural silicates, for instance,) no such effect is produced. This operation has received the name of *flaming*, and any substance thus acted upon is said to become *opaque by flaming*.

3. **Ammonio-phosphate of Soda (Microcosmic Salt)** is ob-

tained by dissolving 16 parts of sal-ammoniac in a very small quantity of boiling water, and mixing with it 100 parts of crystallised phosphate of soda, dissolving the whole with heat, filtering the boiling liquid; during cooling the double salt crystallises. When microcosmic salt is not pure it forms a glass which becomes opaque by cooling. It is then necessary to dissolve it in a small quantity of water and recrystallise it.

It may be collected in large crystals, or in a pulverulent state. The crystals are in general of a suitable size for ordinary assays. Placed on charcoal, and submitted to the blowpipe flame, it bubbles and swells up, giving off ammonia; that which remains after this treatment is an acid phosphate of soda, which fuses readily, and forms on cooling a transparent and colourless glass. As a reagent, it acts principally by its free phosphoric acid; and if the salt be employed in preference to the acid, it is because it is less deliquescent, costs less, and passes readily into the charcoal. By means of microcosmic salt we then ascertain the action of free acids on any substance we may wish to assay. The excess of acid it contains combines with all bases, and forms a class of double salts, more or less fusible, which are examined as to their transparency and colour. In consequence, this flux is used more particularly in the detection of the metallic oxides, most of which impart to it very characteristic colours. This flux exercises on acids a repulsive action. Those which are volatile, sublime; and those which are fixed remain in the mass, dividing the base with the phosphoric acid, or yielding it up entirely; in which case they are suspended in the glass without being dissolved. In this respect, microcosmic salt is a good test for silicates; for by its aid silica is liberated, and appears in the glass as a gelatinous mass.

4. **Nitrate of Potash (Nitre)**, in long and thin crystals, is employed in hastening the oxidation of those substances which do not readily combine with oxygen in the exterior flame. It is used as follows: the point of a crystal is thrust into the fused bead; but in order to prevent the cooling of the latter the crystal is held by a pair of pliers, so that

when the bead begins to cool it may be withdrawn, the bead reheated, and the crystal employed as before, until the desired effect is produced.

5. **Bisulphate of Potash** is employed in the detection of lithia, boracic acid, nitric acid, hydrofluoric acid, bromine, and iodine. It separates baryta and strontia from the earths and metallic oxides.

Professor J. Lawrence Smith[*] has suggested the use of bisulphate of soda as a substitute for the bisulphate of potash in the decomposition of minerals, especially the aluminous minerals. He says that 'bisulphate of potash is still used, to the almost utter exclusion of bisulphate of soda in rendering certain minerals soluble; and it is still recommended as the proper agent to fuse with aluminous minerals, as corundum, emery, &c.

'This subject occupied my attention to a considerable extent when engaged in the preparation of two memoirs on the geology and mineralogy of emery, presented to the French Academy of Sciences in 1850, as well as in some investigations I am now making on the emery from Chester, Massachusetts. In the above researches I had a large number of corundums and emeries to analyse. The powdered minerals were fused with the bisulphate of potash in the usual way, and I found no difficulty in decomposing the minerals; but unfortunately during the operation a double salt of potash and alumina is formed, which is almost insoluble in the water or in the acids; and it is only by a solution of potash that it is first decomposed and afterwards redissolved. There are many disadvantages and delays attendant upon this method, which experience soon exhibits; as the constant deposition of alum, if the solution is not kept quite dilute. I therefore experimented with the bisulphate of soda, knowing that the double salt of alumina and soda was quite soluble, and my results were everything that could be desired; for while the soda salt gives a decomposition at least as complete as the potash salt, the melted mass is very soluble in water, and in the future operations of the analyses there is no embarrassment from a deposit of alum.

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[*] American Journal of Science and Arts.

'The ordinary commercial article is not sufficiently pure for use, and I prepare it from pure carbonate of soda or sulphate of soda that has been purified by recrystallisation. In either instance pure sulphuric acid is added in excess to the salt in a large platinum capsule, and heated over a flame, until the melted mass, when taken up on the end of a glass rod, solidifies quite firmly. The mass is then allowed to cool; moving it over the sides of the capsule will facilitate this operation. When cool it is readily detached from the capsule, is then broken up, and put into a glass stoppered bottle. So far as my experience has yet gone, in almost every instance where we have been in the habit of using bisulphate of potash the bisulphate of soda can be substituted.'

6. **Vitrified Boracic Acid** is used to ascertain the presence of phosphoric acid and small portions of copper in lead alloys. For quantitative analysis, it is generally used to ascertain the quantity of copper contained in a lead ore, and also the amount of copper united with various metals.

7. **Nitrate of Cobalt** in solution ought to be free from arsenic and nickel, and the solution must be moderately strong. It is used as a test for alumina, magnesia, tin, and zinc, by the blowpipe.

8. **Oxalate of Nickel** is used in qualitative examinations for the detection of potash in a salt which also contains soda and lithia.

9. **Oxide of Copper** is employed to detect the presence of hydrochloric acid and chlorine.

10. **Silica** is, with soda, an excellent test for the presence of sulphuric acid; and when in combination with borax or soda, separates tin from copper.

11, 12. **Fluoride of Calcium (Fluor-spar), and Sulphate of Lime (Gypsum).**—These two bodies (deprived of water) are used to indicate the presence of each other. If a small piece of gypsum be ignited in contact with a similar piece of fluor-spar, they soon liquefy at their points of contact; they then combine, and form, by fusing, a colourless and transparent bead of glass, which becomes enamel-white on cooling. Fluoride of calcium is thus employed as a test for gypsum, and *vice versâ*.

It serves, also, when mixed with bisulphate of potash, to detect lithia and boracic acid in their various combinations.

13. **Bone Ashes** are employed in the cupellation of gold and silver. Harkort reduced them to many states of minute division by the processes of sifting and washing. The bones are burned until they become perfectly white, and then freed from any carbonaceous matter that may have adhered to them. This being done, they are pulverised in a mortar, and the finer portions separated by a sieve. The remaining powder is then thrown upon a filter, and treated with boiling water, which extracts the soluble matter. The washing, which is then resorted to, is for procuring the bone ashes of a more uniform degree of fineness. The mass from the filter is mixed with water in a cylindrical glass, allowed to settle for a few minutes and then decanted; the coarser powder is deposited at the bottom of the vessel, while the finer passes over, suspended in the water. By repeated decantations in this way, deposits are obtained of different degrees of fineness; the last, or that which remains longest floating through the liquid, being the finest. The resulting powders must be kept in separate bottles. The coarser ashes are used for cupellation of rich silver ores, and the finer for assaying ores in which only a minute quantity of gold or silver is present.

14. **Proof Lead** is made use of in cupelling argentiferous or auriferous substances; it must be free from silver. Dumas states that the best method of obtaining lead in this desirable state is to decompose the best white-lead by means of charcoal, as it is then impossible for it to contain any other metal.

15. **Tinfoil** is employed to reduce certain peroxides to the state of protoxide. When it is used, a small roll, about ¼ of an inch long, is plunged into the fused button, and heated strongly in the reducing flame: the desired effect is then produced.

16. **Dry Chloride of Silver.**—Herr H. Gericke proposes the employment of this compound in qualitative blowpipe assays. In an elaborate paper on this subject, communicated to the *Chemical Gazette* (vol. xiii. p. 189) he says:—

'Amongst the phenomenà which characterise different bodies before the blowpipe, and serve for their distinction, the colour of the flame is of no small importance. This power of colouring the blowpipe flame is not, however, exhibited by all bodies with sufficient intensity to enable them to be distinguished by it with certainty; and certain substances are consequently usually employed, such as muriatic acid with baryta, strontia and lime, or sulphuric acid, partly to form and partly to set free volatile compounds. By this means, however, although the intensity of the coloration is heightened, its duration is not increased, as these acids, and particularly muriatic acid, evaporate for the most part before they have acted sufficiently, so that the coloration lasts only for a few moments. This defect may be got over by the employment, instead of the volatile muriatic acid, of a chloride, which will retain the chlorine at a high temperature, so that it may only be set free by degrees in small quantities, while the body forming its base may be without action upon the colouring power of the body under investigation. For this purpose chloride of silver appears to be the best, especially as it may readily be prepared in a state of purity. The best plan is to stir it with water into a thick paste, and keep it in a bottle.

'In regard to the action of chloride of silver upon the coloration of the blowpipe flame, I have investigated several compounds of potash, soda, lithia, lime, baryta, strontia, copper, molydenum, arsenic, antimony, and lead, and mixtures of these substances. Chloride of silver, of course, has no action upon borates and phosphates, both of these acids being amongst those which offer the most resistance to the action of heat.

'For a support, I employed first of all platinum wire, but this is soon alloyed by the metallic silver which separates, and thus rendered useless in testing metals. Silver wire is too readily fusible, and also difficult to obtain free from copper, which may give rise to errors when in contact with chloride of silver. For these reasons, iron wire is best fitted for experiments with chloride of silver, as from its cheapness a new piece may be employed for each experiment, while the silver may readily be obtained in the form of chloride from the broken pieces. If the size of the fragment under examination be sufficient, the platinum forceps may be employed.

'The results at which I arrived, by the employment of chloride of silver, in comparison with those obtained without this reagent, are as follows:

'With potash compounds, such as saltpetre, potashes, &c., the flame is decidedly of a darker colour with chloride of silver; and even in ferrocyanide of potassium, which, when treated by itself with the blowpipe, colours the flame blue, the addition of chloride of silver produces a distinct potash coloration.

'The action of chloride of silver upon soda salts is not so favourable; for although with some, as nitrate of soda, common soda, and labradorite, the flame acquires a more intense yellow colour by the addition of chloride of silver, this reagent produces no observable difference with other soda compounds, such as sulphate of soda and analcime. This also applies to the compounds of lithia, some of which give a finer purple-red colour on the addition of chloride of silver, whilst upon others it has no such effect.

'With lime compounds chloride of silver acts favourably upon the colouring power. Thus the addition of chloride of silver to calcareous spar or gypsum (in the reduction flame) gives the flame a more distinct yellowish-red colour, but stilbite gives no coloration either with or without chloride of silver. With fluor-spar the coloration cannot well be observed, as this decrepitates too violently under the blowpipe.

'The action of chloride of silver upon compounds of baryta and strontia is decidedly advantageous, as both the intensity of the coloration and its duration leave nothing to be desired. Siliceous celestine, which, when heated by itself in the forceps, scarcely coloured the flame, immediately produced a permanent red coloration when heated with chloride of silver.

'Although it appears from the preceding statements, that the employment of chloride of silver presents no advantage with some substances, it may be used with good results in the treatment of mixtures of alkalies and earths.

'Thus, with *petalite* alone, the lithia coloration is first produced, and a slight soda coloration is afterwards obtained; whilst with chloride of silver the soda coloration appears very distinctly after that of the lithia. With *lithion mica* alone a very distinct lithia coloration is presented; but in the presence of chloride of silver a colour is first produced which may lead to the conclusion that potash is present, but the lithia coloration is weakened. *Ryacolite*, heated by itself in the blowpipe flame, only gives a distinct soda coloration; but with chloride of silver a slight potash coloration is first produced, and the colour of soda then appears very distinctly; the lime contained in it cannot however be detected by the coloration of the flame.

'Chloride of silver may be employed with still greater advantage with the following metals, but in these cases it is particularly necessary that the operator should become familiar with the colour produced by each individual substance.

'With copper compounds, such as red copper ore, malachite, copper pyrites, sulphate of copper &c., when contained in other minerals so as to be unrecognisable by the eye, the employment of chloride of silver may be of the greatest service, as the smallest quantities of copper, when treated with chloride of silver under the blowpipe, give a continuous and beautiful blue colour to the flame. With chloride of silver the presence of copper may be distinctly ascertained by the blowpipe, even in a solution which is no longer coloured blue by the addition of ammonia.

'The employment of chloride of silver will be equally advantageous with molybdenum, as in this case also the flame gains greatly in intensity. Arsenic, lead, and antimony are already sufficiently characterised, the former by its odour, the two latter by their fumes; but even with these metals chloride of silver may be employed with advantage to render their reactions still more distinct. It is only necessary to observe, that the greenish-blue flame of antimony appears greener and more like that of molybdenum under the influence of chloride of silver.

'Chloride of silver may also be employed with compounds containing several of the above-mentioned metals.

'If *bournonite* be heated in the oxidation flame of the blowpipe, a fine blue flame is first produced, which indicates lead with certainty; if chloride of silver be now applied, copper is also readily shown. The antimony contained in *bournonite* cannot be ascertained by the coloration of the flame; but this may easily be detected upon charcoal, or in a glass tube open at both ends.

'Native molybdate of lead, without chloride of silver, only gives a blue colour to the blowpipe flame; with chloride of silver this blue coloration of the lead comes out more distinctly, but at the same time the tip of the flame, particularly when the reduction flame is employed, appears of a beautiful yellowish-green colour from molybdenum.

'With mixtures of arsenic and copper, or antimony and copper, the flame first acquires a greyish-blue or greenish-blue colour from the oxidation of the arsenic or antimony; the copper may then be very easily detected by chloride of silver. This applies also to mixtures of arsenic and molybdenum, or antimony and molybdenum; with chloride of silver the yellowish-green flame of molybdenum appears distinctly. It will be more difficult to analyse mixtures of arsenic and lead, or antimony and lead, in this manner; and if a compound contain both arsenic and antimony, these two bodies are not to be distinguished with chloride of silver under the blowpipe.

'From these experiments it appears that in blowpipe testing, it is more advantageous to employ chloride of silver, instead of muriatic acid.

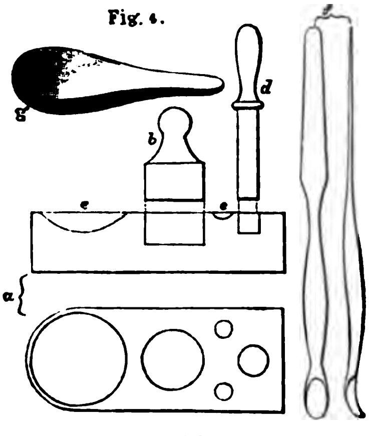
'Chloride of silver is particularly to be recommended in testing metallic alloys for copper. Thus, to test silver for copper, chloride of silver may be applied to the ends of silver wires, and on the application of heat the smallest quantity of copper will furnish the most distinct reaction. This is as sensitive as any of the known copper reactions, and may be performed quickly and easily. In testing metallic alloys for traces of copper, it may be advisable to submit those which contain antimony, zinc, lead, and other volatile metals to roasting, so as to drive off these metals before the addition of chloride of silver.'

### GENERAL ROUTINE OF BLOWPIPE OPERATIONS.

*Size of the Assay.*—The morsel operated on is sufficiently large when the effect of the heat and the fluxes added can be distinctly discerned. The size of the assay-piece generally recommended is much too large; its size ought to be about that of a mustard-seed; that of the flux added, about the size of a hemp-seed. It should in general be previously reduced to fine powder.

**Soda-Paper.**—Mr. Forbes writes as follows in the *Chemical News:* 'As it would be impossible to submit any powdered substance to the direct action of the blowpipe flame without its suffering mechanical loss, some means must be employed for holding the particles together until they are so agglutinated by the heat that no such loss need be apprehended; this is secured by the use of the soda-paper envelope or cornet, as devised by Harkort. For this purpose slips of thin slightly sized writing paper, about 1¼ inch long by 1 inch broad, are steeped in a solution of one part crystallised pure carbonate of soda (free from sulphate) in two parts of water. When dried these are used for forming small cylindrical cornets, by rolling them round the ivory cylinder, fig. 70 *d*, previously described. A hollow is formed to them by folding down a portion of their length on to the end of the cylinder, which is then pressed firmly into the corresponding mould in the blowpipe anvil, and which, upon the withdrawal of the cylinder, serves as a support until they are filled with the assay from the scoop in which the assay and flux have been mixed. After pressing the assay down,

Fig. 70.



the superfluous paper is cut off, leaving only sufficient when folded down upon the contents of the cornet, to form a paper cover to the top similar to the hollow of the cornet. The assay is then ready for placing in a bore in the charcoal, formed by the charcoal borer *c*, fig. 68, and is then submitted to a reducing fusion.'

When a large piece is employed, the experiment consumes so much more time and requires so much more labour than a smaller piece. It is only in reductions that a larger piece may be successfully employed, because in that case the more metal produced, the more readily can its nature be ascertained. Having thus endeavoured to fix the size of the assay, we will now lead our readers to the operations necessary in blowpipe analysis, and in the order in which they are to be performed.

*Firstly.*—The substance is heated in the closed tube, or matrass, over a spirit-lamp. It may, by this treatment, decrepitate, or give off water, or some other volatile substance.

*Secondly.*—It is heated gently on charcoal, by aid of the blowpipe; and, as soon as warm, withdrawn from the heat, and the odour given off ascertained: volatile acids, arsenic, selenium, or sulphur, may be present. The odour thus produced by the oxidising flame must be compared with that produced by the reducing flame; if any difference, it must be carefully noted. Sulphur, selenium, &c., are best detected in the oxidising flame, and arsenic in the reducing flame.

*Thirdly.*—The substance is examined as regards its fusibility. If it be in grains, it is better acted upon on charcoal, notwithstanding its liability to escape on the first insufflation, when not very fusible. But if we can choose the form, it is better to knock off a small splinter, by means of the hammer, and hold it in the flame by the platinum-pointed pincers. A fragment with the most pointed and the thinnest edges ought to be selected. By thus acting, we can always ascertain at a glance if the substance be fusible or not. Infusible substances retain their sharp points and angles, which can be ascertained immediately by means of a microscope. The points are merely rounded in bodies of difficult fusibility, and in substances of easy fusion are rendered globular.

Certain substances, and particularly some minerals, change both aspect and form when exposed to the blowpipe flame, without entering into fusion; some swell up like borax; some of them fuse after tumefaction; others keep in that state without fusion. Some minerals give off a sort of foam on fusing, giving rise to a kind of blebby glass, which, although transparent itself, does not appear so, on account of the multitude of air-bubbles it contains.

This bubbling and tumefaction take place in the greater part of the minerals only at that temperature at which all the water is disengaged; and these ramifications appear to proceed from a new molecular arrangement, produced by the action of heat on the constituent parts of the substance. It cannot be said that the expansion of a particular part of the substance, or its formation into gas, gives rise to this, because it most often happens in those substances which contain no such substance. The minerals which generally give these indications are the double silicates of lime, or alkali and alumina. It sometimes disappears after a few instants, and occasionally lasts as long as the substance is kept in fusion. In the latter case, it seems that the assay takes carbonic acid from the flame, which carbonic acid is transformed by the charcoal into carbonic oxide, and it is that gas which causes the bubbles.

The examination of the comparative fusibility of minerals is of essential importance, as many which consist principally of earths, and contain very little of true metallic oxides, can readily be distinguished by this means. Hence it is that the list given by Rose, of a considerable number of minerals arranged according to their different degrees of fusibility, is of great interest.

Of the minerals which occur most frequently, the following are, when heated between the platinum-points of a forceps in a strong flame, perfectly infusible: *Quartz*, *Corundum*, *Spinel*, *Zeylanite*, *Pleonaste*, *Automolite*, *Gahnite*, *Olivine*, *Cerite*, *Zircon*, *Disthène*, *Cyanite*, *Leucite*, *Talc*, *Gehlenite*, *Anthophyllite*, *Staurolite*, *Allophane*, *Kymophane*, *Gadolinite*.

The following phosphoresce on being heated: *Rutile Titaneisen*, *Tantalite*, *Turquoise*, *Calaite*, *Chondrodite*, *Topaz*.

The following are fusible with difficulty, or only on the edges: *Adularite*, *Tetartine*, *Albite*, *Petalite*, *Labradorite*, *Anorthite*, *Tabular Spar*, *Meerschaum*, *Speckstein*, *Serpentine*, *Epidote*.

The following tumefy on the first application of heat: *Dichroite* (some varieties moderately fusible), *Beryl*, *Emerald*, *Euclase*, *Titanite*, *Sodalite*, *Schwerstein*, *Tungstate of Lime*, *Heavy Spar*, *Sulphate of Baryta*, *Celestine*, *Gypsum*, *Apatite*, *Fluor-spar*.

Amongst fusible minerals are the *Zeolites*, most of which present intumescence when heat is first applied, *Oligoclase*, *Soda Spodumene*, *Spodumene*, which also tumefies, *Meionite*, *Elaolite*, *Nepheline*. *Amphibolite*, the greater part of which effervesce during fusion. The *Pyroxenes*, of which those containing large quantities of magnesia are with difficulty fusible, *Vesuvian*, *Idocrase*, which tumefies on melting, *Orthite*, which boils while fusing, *Wolfram*, *Boracite*, *Datholite*, *Botryolite*, *Tourmaline* and *Axinite*, which swell up when melted, *Amblygonite*, *Lazurstein*, *Hauyn*, *Nosin*, *Eudyalite*, and *Pyrosmalite*.

In the employment of fluxes, it is necessary to continue the blast for a sufficiently long time, because some substances appear infusible at the commencement of the operation, and gradually yield to the influence of the flux, and in about two minutes enter into full fusion. The substance is best added in small quantities, and no new dose must be introduced until the former one is perfectly acted upon, so that at last the glass arrives at that degree of saturation that it can dissolve no more: it is at this particular point the reactions are most vivid and plain. Beads of glass, not so saturated, do not give such decided indications.

Occasionally, in operating with a flux in the reducing flame, it happens that the assay-bead reoxidises during the cooling of the charcoal, and thus the labour of a preceding operation is lost. In order to obviate this inconvenience, the charcoal is turned over, so that the bead may fall in a yet liquid state on some cold body, as a porcelain plate.

When the colour of the bead is so intense that it appears opaque, its transparency can be proved by holding it opposite to the flame of a lamp; the reversed image of the flame can then be seen in the bead, tinged with the colour imparted to the flux by the body under experiment. The globule may also be flattened by a pair of pliers before it cools, or it may be drawn into a thin thread. In either of the last-mentioned cases its colour can readily be ascertained.

Minerals exposed to the exterior and interior flame, either with or without fluxes, present a variety of phenomena, which ought to be carefully noted, and which, collectively, form the result of the assay. The smallest circumstance must not be overlooked, because it may lead us to ascertain the presence of a substance not suspected. It is necessary, in all cases, to make two assays, and compare the separate results; because it sometimes happens that an apparently trivial fact had been overlooked in the first series of operations, which materially conduces to the good result of the experiment.

**Potash, Soda,** and **Lithia** cannot be distinguished with any degree of certainty by the blowpipe; their presence is best ascertained by the wet assay; that is to say, however, with the exception of soda. Potash colours the blowpipe flame bluish; soda, yellow; and lithia, red. These indications, however, will be more fully discussed under the head of *Coloured Flames.*

**Baryta,** alone, is infusible. The hydrate is fusible, but soon becomes a solid crust, on account of its losing water.

*Carbonate of Baryta* ($BaO,CO_2$) fuses very readily into a limpid glass; and, on cooling, takes the appearance of a white enamel. On charcoal it effervesces, and becomes caustic baryta; it then behaves as above stated.

*With borax,* baryta fuses easily into a limpid glass, with a lively effervescence. It becomes opaque by *flaming.*

*With microcosmic salt* it fuses easily, with a brisk effervescence, during which the globule foams and swells; after which it is transformed into a limpid glass.

*With soda* it fuses and sinks into the charcoal.

*With nitrate of cobalt* it produces a bead, which, when hot, is brick-red. It loses this colour by cooling.

**STRONTIA.**—*Alone*, it presents the same phenomena as baryta; as it does also with microcosmic salt and borax.

*Soda* does not dissolve caustic strontia. Carbonate of strontia, mixed with its own volume of soda, fuses into a limpid glass, which becomes enamel-white on cooling.

*With nitrate of cobalt* strontia becomes black, or greyish-black, and does not fuse like baryta.

**LIME**, alone, undergoes no alteration. Carbonate of lime becomes caustic, giving off a very strong light.

*With borax* it readily fuses, giving a limpid glass, which becomes opaque by flaming.

*With microcosmic salt* it fuses in large quantity, giving rise to a limpid glass, which preserves its transparency on cooling.

*Soda* scarcely acts either upon lime or its carbonate, passing into the charcoal, and leaving them unaltered upon its surface.

Acted on by *nitrate of cobalt*, *lime* gives a blackish mass, which is infusible.

**MAGNESIA**, *alone*, undergoes no alteration.

*With borax*, behaves as with lime.

*With microcosmic salt*, fuses readily.

*With soda*, no action.

*With nitrate of cobalt*, after a strong heat, becomes flesh-red; which tint, however, is not well seen until after perfect cooling.

**ALUMINA**, *alone*, does not change.

*With borax*, fuses slowly, and forms a diaphanous glass, which becomes opaque either by cooling or flaming.

*With microcosmic salt*, it forms a transparent glass.

*With soda* it swells a little, forming an infusible compound. The excess of soda is absorbed by the charcoal.

*With nitrate of cobalt* it gives a fine blue colour by a strong blast. This colour is best observed by daylight, and is very characteristic of alumina.

**MOLYBDIC ACID.**—*Alone*, in the open inclined tube, it fuses, giving off a white smoke, which condenses in the form of a white powder on the sides of the tube. Heated on platinum foil, it fuses and smokes. The fused portion is brown, but becomes yellowish and crystalline on cooling. In the reducing flame it becomes blue.

*With borax* it fuses on the platinum wire, forming in the exterior flame a colourless and transparent glass. On charcoal, in the reducing flame, the glass becomes brown, and loses its transparency.

*With microcosmic salt* it fuses on the platinum wire in the exterior flame, producing a transparent glass, which, while hot, is greenish, but which colour it loses on cooling. In the reducing flame, the green becomes opaque, and appears black or deep blue, but by cooling becomes nearly as beautiful a green as that produced by oxide of chromium.

*With soda*, molybdic acid fuses on the platinum wire with effervescence, forming a limpid glass, which becomes milk-white by cooling.

Acted on by soda on charcoal, molybdic acid is absorbed as soon as fused; and by removing the charcoal which has absorbed it, and treating it by washing and grinding, metallic molybdeum may be obtained.

**TUNGSTIC ACID.**—*Alone*, it blackens, but does not fuse in the reducing flame.

*With borax* it fuses readily on the platinum wire, forming a colourless glass in the outer flame, which does not become opaque by flaming. In the reducing flame the glass is yellowish when it contains only a small proportion of acid, and the colour augments in intensity by cooling, becoming perfectly yellow.

*With microcosmic salt*, tungstic acid dissolves, forming in the exterior flame a colourless or slightly yellowish glass. In the reducing flame it becomes a fine blue, more beautiful than that of cobalt. If the acid contains iron, the glass assumes a perfectly different appearance, becoming blood-red.

*Soda* dissolves tungstic acid on the platinum wire, converting it into a transparent and deep yellow glass, which crystallises on cooling, becoming an opaque white or yellow. If tungstic acid be treated on charcoal with a small quantity of soda in the reducing flame, a steel-grey slag is obtained, which, by washing and levigating, furnishes metallic tungsten.

The reactions produced on the various compounds of the commoner metals, by the blowpipe, with and without fluxes, will be given under their respective headings.

**Silica.**—*Alone*, undergoes no change,

*With borax* it fuses slowly and gives a clear glass, of difficult fusion, which is not rendered opaque by flaming.

*Microcosmic salt* dissolves but a very small quantity. The fused glass preserves its transparency after cooling; that which is half fused has but a semitransparency.

*With soda* it fuses, giving rise to brisk effervescence, with the production of a limpid glass.

*With solution of cobalt*, in certain proportions, it takes a faint bluish tint, which becomes black, or deep grey, according to the quantity of cobalt. It is by means of this colour that silica is distinguished from some aluminous substances.

**Sulphur** gives, on burning, the well-known odour which is due to the formation of sulphurous acid. It leaves no residue, when pure, on being heated on platinum foil.

*Compounds of Sulphur with the Metals: Sulphides.*—These bodies may be recognised by the odour of sulphurous acid they exhale when heated on charcoal or in the open tube. When the quantity of sulphur contained in any compound is too small to be detected by the smell, its presence may be ascertained by fusing it with a bead composed of carbonate of soda and silica. The glass, on cooling, takes a brown or reddish-yellow colour, according to the quantity of contained sulphur. This method cannot always be employed, because the associated metals mask the colour; in this case the mineral must be roasted in the open tube, in the upper part of which is placed a piece of Brazil-wood paper. If sulphur be present, the red colour of the paper will disappear. A quantity of sulphur so small as to be imperceptible to the smell, will bleach this test-paper. This method must always be followed in the detection of sulphur in the sulphides of antimony, because it is difficult to ascertain its presence by the smell.

The principal object, however, in view in the examination of the metallic sulphides, is to ascertain the presence of some particular metal, in which case they must be roasted, taking care to observe the precautions pointed out for the roasting of ores for assay by the furnace. The roasting must always be executed in the oxidising flame, and great care taken to apply only a very gentle heat at first, otherwise the assay will fuse, and it will then be impossible to continue the roasting with the sample. Great care must be taken to expel the whole of the sulphur, otherwise no reduced metal can be obtained by the action of soda, as sulphide of sodium forms fusible compounds with most of the metallic sulphides.

**SELENIUM** can be sublimed under the same circumstances as sulphur. The sublimate, if small, is reddish; but if large, is of so deep a colour as to appear black. It gives, when heated in the open air, a strong smell of decayed horseradish. Owing to this peculiar smell, it is very readily distinguished by the blowpipe from all other bodies.

*Selenides.*—With the glass of silica and soda, the selenides behave as the sulphides; but the colour disappears sooner by a long blast than that produced by the sulphides. When a selenide is combined with a sulphide the selenium sublimes as selenium, while the sulphur is disengaged as sulphurous acid. If selenium be found with tellurium, the oxide of tellurium first sublimes, and finally, the selenium is deposited nearest the point heated. Sometimes sulphide of arsenic sublimes with the same appearances as selenium, but never with the same odour.

**SULPHATES.**—The presence of this class of bodies is ascertained in the same manner as sulphur, by means of the glass of soda and silica. The sulphates of the metals proper, when heated with charcoal in the close tube, give off sulphurous acid, which may be detected either by the smell or by its action on Brazil-wood paper. The metals of the alkalies and alkaline earths give no sulphurous acid when treated in this manner.

**Nitrates.**—All the salts of nitric acid deflagrate with carbonaceous matters. This, however, is not characteristic, for the chlorates also possess this property. If any nitrate be heated in the close tube with bisulphate of potash, red fumes of nitrous acid are evolved.

**Bromides**, heated with bisulphate of potash in the closed tube, give off vapours of bromine, which are similar in appearance to those of nitrous acid, but which recall the smell of chlorine. Under the head of *Coloured Flames*, another method of distinguishing bromine will be pointed out.

**Iodides**, acted on by bisulphate of potash, give rise to splendid violet-coloured vapours, which are characteristic. —(Also see *Coloured Flames.*)

**Chlorides**, treated with bisulphate of potash and peroxide of manganese, evolve chlorine, which may be recognised by its peculiar odour and yellowish-green colour.—(For further information, see *Coloured Flames.*)

**Fluorides**, heated with bisulphate of potash, give rise to fluoric acid, which may be distinguished by its power of corroding glass. As fluorine occurs in very small quantities in certain minerals, and as it is rather difficult of detection, full instructions will be given.

In case the mineral is very rich in fluoric acid, it may be mixed with microcosmic salt (previously fused), and heated at the extremity of an open tube, so that part of the current of air feeding the flame can pass into the tube. Aqueous fluoric acid is then formed, which can be recognised by its odour and by the corrosive action it exercises on the tube. If a slip of Brazil-wood paper be held at the opening of the tube, it becomes immediately yellow. On the contrary, when the acid exists but in minute quantity, as in fossils, or where it is combined with weak bases, or with a certain proportion of water, the substance can be heated in the close tube, after the introduction of a piece of moistened Brazil-wood paper. Hydro-fluosilicic acid is liberated by the heat, and a dull ring of silica deposited on the glass, a little above the assay; and lastly, the end of the Brazil-wood paper is turned yellow. Three or four per cent. of fluoric acid can be detected in this manner.

**Phosphates.**—The following is the method recommended by Berzelius for the detection of phosphoric acid. 'The substance to be assayed is dissolved in boracic acid, and, when a good fusion is effected, a piece of fine steel wire, a little longer than the diameter of the bead, is forced into it, and the whole then exposed to a good reducing flame. The iron is oxidised at the expense of the phosphoric acid, causing the formation of a borate of the oxide of iron and phosphuret of iron, which fuses at a sufficiently high temperature. The bead is then taken from the charcoal, enveloped in a piece of paper, and struck lightly with a hammer, by which means the phosphuret of iron is separated from the surrounding flux. It exists as a metallic-looking button, attractable by the magnet, frangible on the anvil, the fracture having the colour of iron. If the substance under assay contained no phosphoric acid, the iron wire will keep its form and metallic lustre, excepting at the ends, where it will be oxidated and burnt. The substance to be assayed ought not to contain sulphuric acid, arsenic acid, or any metallic oxides reducible by iron.'

**Hydrates.**—The presence of water in these substances can be ascertained by heating them in the close tube. If any water be present, it will vaporise and condense on the coolest portions of the tube.

**Silicates.**—These compounds of silica with bases are decomposed by fusion with microcosmic salt, the silica being set at liberty, and the base combining with the phosphoric acid. When but a small quantity of microcosmic salt is employed, it often happens that the silica swells at the moment of decomposition, absorbing the liquefied mass. By adding a large quantity of the flux, the whole can be converted into a globule, which retains in suspension the semi-transparent tumefied silica. This can best be perceived when the glass is in a state of ignition.

**Coloured Flames.**—There are a great number of substances best detected by the colours they impart to the flame of the blowpipe. Indeed, so important is this point, that it has been thought advisable to collect all the facts known on this subject into one place, rather than scatter them over the work. These experiments are best made in a dark room, and with a very small flame.*

### BLUE FLAMES.

| | |
|---|---|
| Large intense blue | Chloride of copper. |
| Pale clear blue | Lead. |
| Light blue | Arsenic. |
| Blue | Selenium. |
| Greenish blue | Antimony. |
| Blue mixed with green | Bromide of copper. |

### GREEN FLAMES.

| | |
|---|---|
| Intense emerald green | Thallium. |
| Very dark green, feeble | Ammonia. |
| Dark green | Boracic acid. |
| Dark green | Iron wire. |
| Full green | Tellurium. |
| Full green | Copper. |
| Emerald green, mixed with blue | Iodide of copper.<br>Bromide of copper. |
| Pale green | Phosphoric acid. |
| Very pale apple green | Baryta. |
| Intense whitish green | Zinc. |
| Bluish green | Binoxide of tin. |

### YELLOW FLAMES.

| | |
|---|---|
| Intense greenish yellow | Soda. |
| Feeble brownish yellow | Water. |

### RED FLAMES.

| | |
|---|---|
| Intense crimson | Lithia. |
| Red | Strontia. |
| Reddish purple | Lime. |
| Violet | Potash. |

*Chlorine*, combined with copper, gives an intense blue flame. This phenomenon may be produced as follows:—Take a piece of thin brass wire, and bend one end of it several times upon itself; place upon this some microcosmic salt, and fuse it until it has acquired a green colour. Then add to it the substance suspected to contain chlorine, and place it in the oxidising flame, just at the point of the blue flame; if any chloride be present a splendid blue colour will be produced.

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* Griffin's *Blowpipe Analysis*, page 148.

*Lead.*—The blue colour produced by this metal is readily obtained. Fragments of a mineral must be held in the tongs, and powder may be assayed on charcoal.

*Arsenic*, in the metallic state, gives rise to a light blue flame.

*Selenium* and *Antimony*, when treated in the same manner, afford characteristic flames.

*Bromine.*—If any substance containing bromine be placed in a bead of fused microcosmic salt on the brass wire, and then in the oxidising flame, a bright blue flame with emerald green edges will be produced.

*Boracic Acid.*—The following is Dr. Turner's process for the detection of boracic acid. 'The substance is to be mixed with a flux composed 1 part of fluor-spar and 4½ parts of bisulphate of potash. This mixture is to be made to adhere to the moistened end of a platinum wire, and held at the point of the blue flame; at the instant of fusion, a dark green flame will be produced. It may also be produced by merely dipping the mineral in sulphuric acid, and exposing it to the blowpipe blast. In case a very small quantity of boracic acid is contained in a mineral, the following process may be employed:—The substance must be fused with carbonate of potash on charcoal, moistened with a drop or two of sulphuric acid, and then a few drops of alcohol: the latter will burn with a green flame when exposed to the flame of the blowpipe.

*Tellurium.*—The peculiar flame given by this metal is produced by heating a portion of its oxide on charcoal in the reducing flame.

*Copper.*—All the compounds of copper, except those in which bromine and chlorine enter, give a beautiful green flame. The soluble salts give it *per se*, but the insoluble require moistening with sulphuric acid.

*Iodine and Copper.*—To the bead of microcosmic salt on the brass wire add any compound containing iodine, and a bright green flame will be produced when the mass is heated in the oxidising flame.

*Phosphoric Acid.*—The phosphates, when moistened with sulphuric acid, give a light green tint to the outer flame.

*Baryta.*—The soluble salts of baryta give a light green colour to the outer flame when moistened with water.

*Zinc*, when exposed to the blowpipe flame, burns with an intense whitish-green light.

*Soda.*—Any salt of soda, or substance containing soda, being exposed to the outer flame, gives a brush of intensely coloured flame, of a fine amber or greenish-yellow.

*Water.*—Certain minerals containing water give a feeble yellowish tint to the flame.

*Strontia.*—All the salts of this substance which are soluble in water give a crimson tint to the flame, which does not endure after the substance is fused. Carbonate of strontia must be moistened with hydrochloric acid, and sulphate of strontia must be reduced to the state of sulphide by ignition with charcoal; it must then be moistened with hydrochloric acid; after which treatment it will exhibit the characteristic flame.

*Lithia.*—All that has been said of strontia applies to lithia, with the remarkable exception, that the coloured flame given by lithia is permanent, whilst that afforded by strontia is evanescent.

*Lime* acts as strontia.

*Potash*, treated as soda, gives a purplish light; but the reactions of potash and soda with oxide of cobalt are the best tests of their presence, combined with the peculiar light afforded by soda.

# CHAPTER VIII.

## VOLUMETRIC ANALYSIS.

The main feature of volumetry is not so much analysis in the proper sense of the term, as the quantitative determination of one principal constituent of a substance.

This determination is done by means of solutions, containing a certain quantity of reagents in a certain volume, which is called a standard solution, the quantity used of such solution being measured by graduated tubes (burettes, pipettes, &c.).

The reaction of a volumetric analysis can be of three different kinds, according to the reagent used and to the substance to be determined.

1. The substance to be analysed being an acid or a base, it can be saturated by a suitable standard solution (saturation-analysis, used for acids, potash, soda, &c.).

2. The substance to be assayed may be precipitated by the standard solution, and the completion of the process is observed when no further precipitate occurs (precipitation-analysis, e.g. Pelouze's copper assay, Gay-Lussac's silver-assay).

3. The substance to be determined becomes, by the standard solution, either oxidised or reduced, and by the performance of this process certain colours will appear or disappear, from which the completion of the process is to be observed (oxidation or reduction-analysis, e.g. Schwarz's copper assay).

These processes of volumetric analysis are frequently used in assaying.

The principle of volumetric analysis may be fully explained by the following examples given by Fresenius.*

'Suppose we have prepared a solution of chloride of sodium of such a strength that 100 c. c. will exactly precipitate 1 grm. silver from its solution in nitric acid, we can use it to estimate unknown quantities of silver. Let us imagine, for instance, we have an alloy of silver and copper in unknown proportion, we dissolve 1 grm. in nitric acid, and add to the solution our solution of chloride of sodium, drop by drop, until the whole of the silver is thrown down, and an additional drop fails to produce a further precipitate. The amount of silver present may now be calculated from the amount of solution of chloride of sodium used. Thus, supposing we have used 80 c. c., the amount of silver present in the alloy is 80 per cent.; since, as 100 c. c. of the solution of chloride of sodium will throw down 1 grm. of pure silver (i.e. of 100 per cent.), it follows that every c. c. of the chloride of sodium solution corresponds to 1 per cent. of silver.

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* Fresenius's *Quantitative Analysis*, fourth edition, page 76.

'Another example. It is well known that iodine and sulphuretted hydrogen cannot exist together: whenever these two substances are brought in contact, decomposition immediately ensues, the hydrogen separating from the sulphur and combining with the iodine ($I + HS = HI + S$). Hydriodic acid exercises no action on starch-paste, whereas the least trace of free iodine colours it blue. Now, if we prepare a solution of iodine (in iodide of potassium) containing in 100 c. c. 0·7470 grm. iodine, we may with this decompose exactly 0·1 grm. sulphuretted hydrogen; for 17 : 127 :: 0·1 : 0·7470.

'Let us suppose, then, we have before us a fluid containing an unknown amount of sulphuretted hydrogen, which it is our intention to determine. We add to it a little starch-paste, and then, drop by drop, our solution of iodine, until a persistent blue coloration of the fluid indicates the formation of iodide of starch, and hence the complete decomposition of the sulphuretted hydrogen. The amount of the latter originally present in the fluid may now be readily calculated from the amount of solution of iodine used. Say, for instance, we have used 50 c. c. of iodine solution, the fluid contained originally 0·05 sulphuretted hydrogen; since, as we have seen, 100 c. c. of our iodine solution will decompose exactly 0·1 grm. of that body.

'Solutions of accurately known composition or strength, used for the purposes of volumetric analysis, are called *standard solutions*. They may be prepared in two ways, viz. (*a*) by dissolving a weighed quantity of a substance in a definite volume of fluid; or (*b*) by first preparing a suitably concentrated solution of the reagent required, and then determining its exact strength by a series of experiments made with it upon weighed quantities of the body for the determination of which it is intended to be used.

'In the preparation of standard solutions by method *a*, a certain definite strength is adopted once for all, which is usually based upon the principle of an exact correspondence between the number of grammes of the reagent contained in a litre of the fluid, and the equivalent number of the reagent (H=1). In the case of standard solutions prepared by method *b*, this may also be easily done, by diluting to the required degree the still somewhat too concentrated solution, after having accurately determined its strength; however, as a rule, this latter process is only resorted to in technical analyses, where it is desirable to avoid all calculation. Fluids which contain the equivalent number of grammes of a substance in 1 litre, are called *normal solutions*; those which contain $\frac{1}{10}$ of this quantity, *decinormal solutions*.

'The determination of a standard solution intended to be used for volumetric analysis is obviously a most important operation; since any error in this will, of course, necessarily falsify every analysis made with it. In scientific and accurate researches it is, therefore, always advisable, whenever practicable, to examine the standard solution—no matter whether prepared by method *a* or by method *b*, with subsequent dilution to the required degree—by experimenting with it upon accurately weighed quantities of the body for the determination of which it is to be used.

'In the previous remarks no difference has been made between fluids of known composition and those of known power; and this has hitherto been usual. But by accepting the two expressions as synonymous, we take for granted that a fluid exercises a chemical action exactly corresponding to the amount of dissolved substance it contains; that, for instance, a solution of chloride of sodium containing 1 eq. NaCl will precipitate exactly 1 eq. silver. This presumption; however, is very often not absolutely correct. In such cases, of course, it is not merely advisable, but even absolutely necessary, to determine the strength of the fluid by experiment, although the amount of the reagent it contains may be exactly known; for the power of the fluid can be inferred from its composition only approximately, and not with perfect exactness. If a standard solution keeps unaltered, this is a great advantage, as it dispenses with the necessity of determining its strength before every fresh analysis.

'That particular change in the fluid, operated upon by means of a standard solution, which marks the completion of the intended decomposition, is termed the FINAL REACTION. This consists either in a *change of colour*, as is the case when a solution of permanganate of potash acts upon an acidified solution of protoxide of iron, or a solution of iodine upon a solution of sulphuretted hydrogen mixed with starch paste; or in the *cessation of the formation of a precipitate* upon further addition of the standard solution, as is the case when a standard solution of chloride of sodium is used to precipitate silver from its solution in nitric acid; or in *incipient precipitation*, as is the case when a standard solution of silver is added to a solution of hydrocyanic acid mixed with an alkali; or in *a change in the action of the examined fluid upon a particular reagent*, as is the case when a solution of arsenite of soda is added, drop by drop, to a solution of chloride of lime, until the mixture no longer imparts a blue tint to paper moistened with iodide of potassium and starch-paste, &c.

'The more sensitive a final reaction is, and the more readily, positively, and rapidly it manifests itself, the better is it calculated to serve as the basis of a volumetric method. In cases where it is an object of great importance to ascertain with the greatest practicable precision the exact moment when the reaction is completed, the analyst may sometimes prepare, besides the actual standard solution, another, ten times more dilute, and use the latter to finish the process carried nearly to completion with the former.

'But a good final reaction is not of itself sufficient to afford a safe basis for a good volumetric method; this requires, as the first and most indispensable condition, that the particular decomposition which constitutes the leading point of the analytical process should—at least under certain known circumstances—remain unalterably the same. Wherever this is not the case—where the action varies with the greater or less degree of concentration of the fluid, or according as there may be a little more or less free acid present; or according to the greater or less rapidity of action of the standard solution; or where a precipitate formed in the course of the process has not the same composition throughout the operation—the basis of the volumetric method is fallacious, and the method itself, therefore, of no value.

'When the new system of volumetric analysis first began to find favour with chemists, a great many volumetric methods were proposed, based simply upon some final reaction, without a careful study of the decomposition involved; the result has been a superabundant crop of new volumetric methods, of which a great many are totally fallacious and useless.'

The only condition on which the volumetric system of analysis can be carried on successfully is, that the greatest care is exercised with respect to the graduation of the measuring instruments, and the strength and purity of the standard solutions. A very slight error in the analytical process becomes considerably magnified when calculated for pounds, hundredweights, or tons of the substance tested. The end of the operation in this method of analysis is in all cases made apparent to the eye. (Sutton.)

Though our countrymen *Faraday*, *Penny*, *Ure*, *Griffin*, *Scott*, *Sutton*, and others, have rendered great services to the development of volumetry, the German and French chemists, *Liebig*, *Bunsen*, *Mohr*, *Gay-Lussac*, *Descroillez*, have been the founders of it, and we are chiefly indebted to *Mohr* for the present high perfection of volumetry, which, saving a large amount of time and labour, as compared with the older methods of research, is, in many instances, to be preferred to the latter methods.

In the *Annal. der Chemie und Pharmacie*, cxvi. p. 128, Dr. Mohr, of Coblentz, propounded a problem in volumetric analysis, and invited those interested in the subject to solve it. The first who did so was to receive one of Dr. Mohr's burettes as a prize. Dr. Mohr asserted at the same time that he had himself already solved it.

The problem was,—'To perform quantitative determinations without the use of weights, with volumetric solutions of unknown strength, and the strength of which must not be ascertained and regulated.' Dr. Mohr received a number of communications, in which, however, some condition was violated; either a weight of some kind was used, or the strength of the test liquid was indirectly determined by saturated solutions of chloride of sodium (as in Liebig's urea test), or in some other way.

The problem was first solved by Dr. Pauli, of the Union Alkali Works, St. Helen's, Lancashire, in December 1860, in the following way. In one of the pans of a balance is put a piece of chemically-pure carbonate of soda, and in the other an equal weight of an undetermined soda, and both are measured by an acid of unknown strength. Suppose that in the first case 15 cubic centimètres, and in the second 11 cubic centimètres have been used, then 15 : 11 :: 100 : 73·33. The soda, therefore, contains 73·33 per cent. carbonate of soda.

The solution of the question, for which Dr. Pauli only cites a special case, is generally this:—

Equal portions of the pure substance to be determined and the impure, are to be weighed off, one against the other, and are then to be measured with the same fluid; then the cubic centimètres used for the pure substance represent 100 per cent., and the other number proportionately less. Suppose, for example, that it is proposed to determine the iron in an iron ore. A piece of pure iron wire is placed in the scale of a balance, and is exactly counterpoised by means of the powdered ore; both are then brought in solution as protoxide in the usual way, and are then treated with permanganate of unknown strength. If oxide of iron is to be determined, pure oxide must be used instead of the wire. If potashes are to be examined, pure carbonate of potash (recently heated to redness) should be used. It would appear from this, that for every analysis the strength must be determined by the pure substance. This, however, is easily avoided if we put a sixpence into the scale-pan, and weigh with this both the pure and impure substance. The number of cubic centimètres of the fluid holds good as long as the same fluid and the same sixpence are used, and this number may be marked upon the bottle as expressing 100 per cent. for the same substance.

This method is capable of universal application, and eliminates possible errors in weights and variations of temperature. It is only necessary that the substance to be determined should be available in the pure state. But how is the problem to be solved, if it remains as before, but with the further condition, 'when the substance to be determined is not available in a pure state?' Dr. Mohr received two other solutions of the problem from Dr. Hiller and Herr Dietrich, both students in Heidelberg. Dr. Hiller has solved the question, even with the condition that the pure substances should not be available, and in the same way as Dr. Mohr had already done it. For example,—if no chemically pure peroxide of iron can be obtained for a manganese determination, according to Dr. Hiller, pure permanganate, or pure bichromate of potash, can be employed to weigh off the manganese; both are then converted by distillation with hydrochloric acid into chlorine, and then into iodine, and both fluids are then to be determined with the same unknown solution of hyposulphate of soda. Instead of using pure permanganate it would be better perhaps to use pure iodine to counterpoise the manganese, then to dissolve the iodine in iodide of potassium, and proceed as before. We have now to convert the value of the iodine into the value of an equal weight of pure peroxide of manganese, and express it in the cubic centimètres of hyposulphite used. As 43·57 $MnO_2$ set 127 iodine at liberty, therefore any given weight of $MnO_2$ would set at liberty $\frac{127}{43 \cdot 57}$, or 2·914 times as much iodine as is employed to counterbalance it. We have, therefore, to multiply the number of cubic centimètres of hyposulphite which have been used for a quantity of iodine equal to the manganese by 2·915, and then to proceed as if pure $MnO_2$ and common manganese had been weighed together.

If pure carbonate of soda cannot be obtained, pure carbonate of lime may be used; the cubic centimètres of the acid used must be multiplied by $\frac{50}{53}$, that is, 1 atom of carbonate of lime divided by an atom of carbonate of soda.

## STANDARD SOLUTIONS.

We will only add here the preparation of permanganate of potash, and will give the preparation of other solutions when describing the processes for which they are used.

The following description is condensed from Mr. Sutton's excellent work on the subject.[*]

The pure permanganate may be obtained very generally of the dealers in pure chemicals, but should it not be procurable when required, or the expense be too great, the solution may be prepared as follows:—

Ten parts of caustic potash and 7 of chlorate of potash are fused in a Hessian crucible, then 8 parts of finely powdered peroxide of manganese added, and the whole well mixed with an iron rod; the crucible is kept at a dull red heat, and the contents stirred until, from the dissipation of the water, the mass loses its pasty state and becomes somewhat friable; continue the dull red heat, breaking the mass from the sides of the crucible and mixing altogether for a few minutes, then empty the contents into a clean copper or iron dish. When cool, it is to be coarsely powdered, put into a large flask or porcelain dish, and 20 or 30 times its weight of boiling water poured over it, then kept boiling gently until the solution assumes a deep purple rose colour. When the precipitated oxide of manganese has somewhat settled, the solution may be decanted into a large green glass bottle, and further diluted with the washings of the residue in the dish or flask to about the strength required for analysis. The solution so prepared contains a large quantity of alkali, and is constantly undergoing a slight change owing to its containing a portion of manganate of potash which slowly decomposes with precipitation of oxide of manganese. If the excess of caustic potash is saturated by an acid, the solution is far more stable. Mülder, therefore, recommends that a stream of carbonic acid should be passed through the solution, frequently shaking it, until the potash is saturated; an excess of acid does no harm; sulphuric acid may also be used for the same purpose, but is not so recommendable.

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[*] '*Volumetric Analysis,*' p. 84, *et seq.*

When the liquid thus treated has thoroughly settled, a portion may be decanted (not filtered through paper) into a convenient sized bottle for laboratory use.

A very useful form of bottle for preserving it is the ordinary wash-bottle, or any common bottle fitted with the same arrangement of tubes. Burettes can then be filled with the solution without its frothing, and as the tube which enters the liquid does not reach the bottom of the bottle, the sediment, if any, is not disturbed; another advantage is, that the solution does not come into contact with the cork, nor can any dust enter: the blowing tube may be closed by a very small cork.

A solution prepared and kept as here directed will generally preserve its strength unaltered for six months.

### *The Instruments and Apparatus.*

*The Burette*—or graduated tube for delivering the standard solution, may be obtained in a great many forms, under the names of their respective inventors, such as Mohr, Gay-Lussac, Binks, &c., but as some of these possess a decided superiority over others, it is not quite a matter of indifference which is used, and therefore a slight description of them may not be out of place here. The burette, with india-rubber tube and clip, contrived by Dr. Frederic Mohr of Coblentz, shown in Figs. 71 and 72, has the preference above all others for general purposes.

The advantages possessed by this instrument are, that its constant upright position enables the operator at once to read off the number of degrees of test solution used for any analysis. The quantity of fluid to be delivered can be regulated to the greatest nicety by the pressure of the thumb and finger on the spring clip, and the instrument not being held in the hand, there is no chance of increasing the bulk of the fluid by the heat of the body, and thus leading to incorrect measurement, as is the case with Binks' or Gay-Lussac's form of instrument. The principal disadvantage, however, of these two latter forms of burette is, that a correct reading can only be obtained by placing them in an upright

FIG. 71.

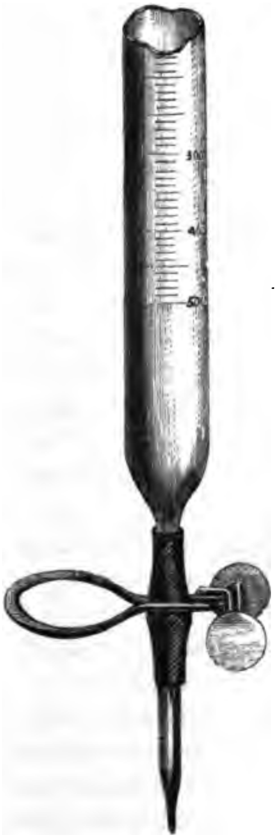
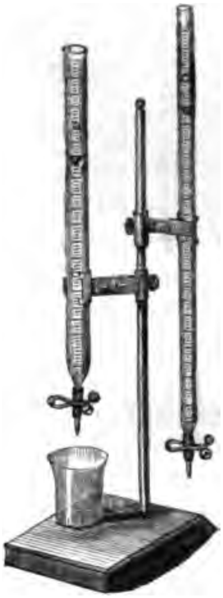


FIG. 72.



position, and allowing the fluid to find its perfect level. The preference should therefore, unhesitatingly, be given to Dr. Mohr's burette, wherever it can be used; the greatest drawback to it is, that it cannot be used for permanganate of potash in consequence of its india-rubber tube, which decomposes the solution.

We are again indebted to Dr. Mohr for another form of instrument to overcome this difficulty, viz., the foot burette, with india-rubber ball, shown in Fig. 73.

Fig. 73.

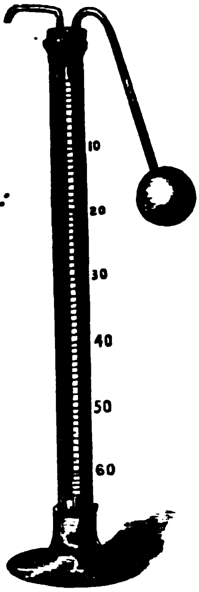
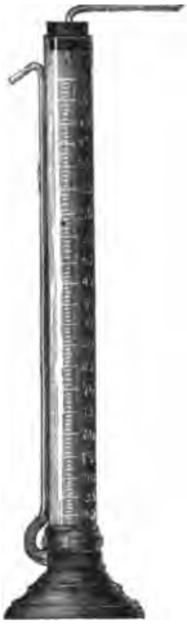


Fig. 74.



The flow of liquid from the exit tube can be regulated to a great nicety by pressure upon the elastic ball, which is of the ordinary kind sold for children, and has two openings, one cemented to the tube with shellac, and the other at the side, over which the thumb is placed when pressed, and on the removal of which it refills itself with air.

Gay-Lussac's burette, supported in a wooden foot, may be used instead of the above form, by inserting a good fitting cork into the open end, through which a small tube bent at right angles is passed. If the burette is held in the right hand, slightly inclined towards the beaker or flask into which the fluid is to be measured, and the mouth applied to the tube, any portion of the solution may be emptied out by the pressure of the breath, and the disadvantage of holding the instrument in an horizontal position, to the great danger of spilling the contents, is avoided; at the same time the beaker or flask can be held in the left hand and shaken so as to mix the fluids, and by this means the end of the operation more accurately determined.

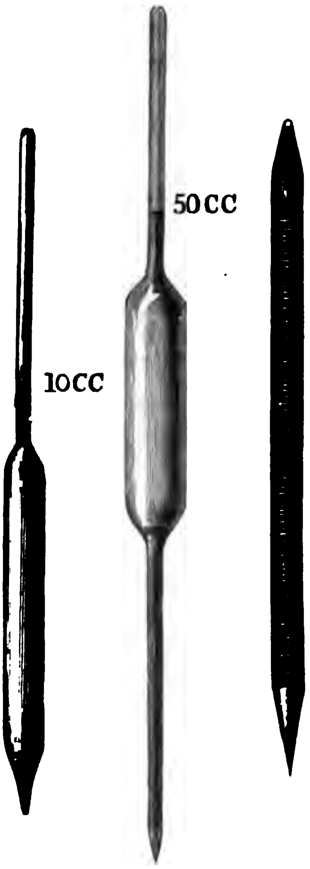
Fig. 74 will show the arrangement here described.

*The Pipette.*—The pipettes used in volumetric analysis are of two kinds, viz., those which deliver one certain quantity only, and those which are graduated so as to deliver various quantities at the discretion of the analyst. In the former kind, or whole pipette, the graduation may be of three kinds, namely, 1st, in which the fluid is suffered to run out by its own momentum only. 2nd, in which it is blown out by the breath. 3rd, in which it is allowed to run out to a definite mark. Of these methods the last is preferable in point of accuracy, and should therefore be adopted if possible. The next best form is that in which the liquid flows out by its own momentum, but in this case the last few drops empty themselves very slowly; but if the lower end of the pipette be touched against the beaker or other vessel into which the fluid is poured, the flow is hastened considerably, and in graduating the pipette, it is preferable to do it on this plan.

In both the whole and graduated pipettes, the upper end is narrowed to about $\frac{1}{8}$ inch, so that the pressure of the moistened finger is sufficient to arrest the flow at any point.

Fig. 75 shows two whole pipettes, one of small and the other of large capacity, and also a graduated pipette of medium size.

Fig. 75.



*The Measuring Flasks.*—These indispensable instruments are made of various capacities; they serve to mix up standard solutions to a given volume, and also for the subdivision of the substance to be tested by means of the pipettes, and are in many ways most convenient. They should be tolerably wide at the mouth, and have a well-ground glass stopper, and the graduation line should fall just below the middle of the neck, so as to allow room for shaking up the fluid.

Colorimetric Analysis is also used in assaying. It is based upon the fact that a coloured solution appears the more intense the more of the colouring substance it contains.

If, therefore, a solution containing a certain amount of a substance and being in consequence of a certain intensity of colour, is prepared, it will be possible to obtain the solution under assay of an equal intensity of colour by appropriate dilution.

By measuring the volume of the assay solution and taking into consideration the amount of the standard solution, the quantity of the substance contained in the assay solution may readily be calculated.

# CHAPTER IX.

## THE ASSAY OF IRON.

THE ores of iron, properly so called, always contain the metal in the oxidised state, and in various degrees of purity.

These ores are the following:—

1. **MAGNETIC IRON ORE** ($Fe_3O_4 = Fe_2O_3 + FeO$). When pure, it contains 72 per cent. Fe.

2. **RED HÆMATITE**, *anhydrous sesquioxide of iron* ($Fe_2O_3$). When pure it contains 70 per. cent. Fe. It occurs in different varieties.

3. **BROWN HÆMATITE**, *brown iron ore*, *limonite*, *hydrated sesquioxide of iron* ($2Fe_2O_3,3HO$). When pure it contains 59·89% Fe, and 14·44 of water. It also occurs in many varieties, *Bog iron ore* being one of them.

4. **SPATHIC CARBONATE**, *sparry iron ore*, *crystallised carbonate of protoxide of iron* ($FeO,CO_2$). When pure it contains 48·275 Fe. It generally contains some percentages, (2 to 15) of carbonate of protoxide of manganese, and carbonate of magnesia, frequently also some carbonate of zinc.

5. **ARGILLACEOUS IRON ORES**, *clay or clay-band ironstone*, *impure earthy carbonate of protoxide of iron.* This mineral sometimes resembles compact limestone, sometimes greyish hardened clay. Its great specific gravity, its effervescing on the addition of an acid, and acquiring a brown-red colour on roasting, are sufficient means of identifying it.

The following is the result of an analysis of this class of ore by the Author; the specimen was from Ireland, county Leitrim:—

| | |
|---|---|
| Protoxide of iron | 51·653 |
| Peroxide of iron | 3·742 |
| Oxide of manganese | ·976 |
| Alumina | 1·849 |
| Magnesia | ·284 |
| Lime | ·410 |
| Potash | ·274 |
| Soda | ·372 |
| Sulphur | ·214 |
| Phosphoric acid | ·284 |
| Carbonic acid | 31·142 |
| Silica | 6·640 |
| Carbonaceous matter and loss | 2·160 |
| | 100·000 |

Blackband is a combustible schistose variety of this ore. The following analysis is also by the Author:—

| | |
|---|---|
| Protoxide of iron | 20·924 |
| Peroxide of iron | ·741 |
| Oxide of manganese | 1·742 |
| Alumina | 14·974 |
| Magnesia | ·987 |
| Lime | ·881 |
| Potash<br>Soda | traces |
| Phosphoric acid | ·114 |
| Silica | 26·179 |
| Sulphur | ·098 |
| Carbonic acid | 14·000 |
| Carbonaceous matter | 16·940 |
| Water and loss | 2·420 |
| | 100·000 |

6. **Red Siliceous Iron** (silica, water, peroxide and protoxide of iron). This mineral occurs only in rare cases in sufficiently large quantities to be used for iron smelting.

Besides these iron ores the following substances, containing iron and used as fluxes, require assaying: Granite, Chlorite, Basalt, Pyrozene, Amphibole, and also some kind of slags (finery cinder, tap cinder, etc.).

## A. THE ASSAY OF IRON IN THE DRY WAY.

It has been already mentioned that iron ores very seldom occur in a pure state, and the ores may be arranged for their assay in the dry way (and also for smelting) into five classes.

1. Iron ores containing silica, lime, and another base, which ores are fusible, *per se*.
2. Iron ores containing predominantly silica.
3. „ „ „ „ lime.
4. „ „ „ „ alumina.
5. Iron ores containing a large amount of magnesia; these ores are most difficultly rendered fluid.

The flux used for assaying (and also melting) varies according to the nature of the predominant compound, and the quantity used according to the amount of that compound.

If the composition of the ore is known, it is easy to ascertain the amount of flux necessary to form a slag with the bases or silica present; in most cases an extra quantity of the flux should be added, in order to produce a sufficient volume of slag to cover the button.

According to Dr. Percy,* blast-furnace cinder of the following formula may be taken as a type of the kind of slag desirable:—

$$Al_2O_3,\ SiO_3\ 2\ (3CaO,\ SiO_3).$$

Its approximate composition per cent. is as under:

| | | | |
|---|---|---|---|
| Silica | 38 | or about | 2½ parts |
| Alumina | 15 | | 1 „ |
| Lime | 47 | | 3 „ |

The following mixtures of various fluxes, when fused, produce a slag which may be regarded as approximating to the above composition:

| | | | | | | |
|---|---|---|---|---|---|---|
| Quartz | 1 | | | 1·92 | = | 36·5 per cent. |
| China clay | 2 | Silica | 0·92 | | | |
| | | Alumina | 0·82 | 0·82 | | 15·5 „ |
| Lime | 2½ | | | 2·5 | | 48·0 „ |
| Glass | 2½ | Silica | | 1·75 | = | 35 „ |
| | | Materials = to Alumina† | | 0·75 | | 15 „ |
| Lime | 2½ | | | 2·5 | | 50 „ |
| Shale or fire clay | 3 | Silica | | 1·8 | = | 35 „ |
| | | Alumina | | 0·9 | | 17 „ |
| Lime | 2½ | | | 2·5 | | 48 „ |

According to *Bodemann* a compound of 56% silica, 30% lime, and 14% alumina forms a slag most easily rendered fluid, but as it is found that this slag itself is not sufficiently fusible in a small assaying furnace (air furnace), an addition of fluor-spar is given to the mixture, and in some cases (the iron ore being very difficultly rendered fluid), some borax is added, or a mixture of borax and fluor-spar.

---

* Percy's *Metallurgy*, p. 240.

† 30% say of alkalies, lime, etc., on account of their fusibility, are taken as equivalent to so much alumina.

An exact knowledge of the mineralogical properties of the iron ores, and a due experience, will enable the assayer to properly adjust the fluxes without resorting to an analysis to find what amount of silica and bases are present.

In some iron works of Germany the following proportions of fluxes are used :—

| | | | | | |
|---|---|---|---|---|---|
| For magnetic ore, red hæmatite (very rich) . . . | 5 to 20% | chalk | and | 25% | fluorspar |
| „ argillaceous brown iron ore | 20 to 40 „ | „ | „ | 30 to 40 „ | „ |
| „ bog iron . . . . | 50 „ | „ | „ | 50 „ | „ |
| „ spathose iron ore . . . | 10 to 15 „ | china clay | „ | 20 to 25 „ | „ |
| „ finery cinder . . . | 20 to 25 „ | chalk | „ | 20 to 25 „ | „ |

Air-furnaces are best adapted for assaying iron ores where many assays are required. And naked crucibles, either of clay or blacklead, or crucibles lined with charcoal, are employed; the latter are preferable.

The button of metal does not adhere to naked pots, but the slag adheres very strongly; so much so, that it cannot be detached with any degree of accuracy for weighing (which in some of M. Berthier's processes is of importance). Black-lead pots allow neither the slag nor button to adhere, but the former dissolves much argillaceous matter from the pot, so that its weight is greatly increased, and the assay cannot be verified. In naked crucibles, charcoal must always be added to the assay, to reduce the oxide of iron; in which case, if an excess be added, it prevents the button from completely forming, so that globules remain in the slag (with care this may, however, be avoided). Neither do naked crucibles resist the fire as well as those lined with charcoal, because the lining supports the sides when they soften. The charcoal lining also allows the assay to be finished without adding any re-agent to the ore; the button can be readily taken out, because it does not adhere to the charcoal; and lastly, the earthy matters in the ore, which have formed a slag, may be collected and weighed; or, if we have added any flux to the ore, the total weight can also be ascertained.

After having finely powdered and sifted the iron ore, a determinate weight must be taken (200 grains is the most convenient quantity in ordinary cases); a certain weight of the requisite flux must be well mixed with the ore in a mortar, and the whole placed in the crucible (which must be lined with charcoal), in which it is gently pressed by a pestle or other appropriate instrument, and covered with a slight layer of fluor-spar. The crucible is then filled with successive layers of charcoal powder, slightly moistened with water; a cover luted on it, and the whole placed in the fire. The fire is allowed to burn gently for about an hour, and the heat raised to whiteness for about the same time, or a little longer; the crucibles are taken out, allowed to cool, and the button and flux removed.

The whole fused mass is then weighed, and then the button of metal carefully separated from the flux, and weighed; sometimes small globules of iron are found adhering to the flux, in which case they must be removed, and added to the button before weighing. Even when very small, their removal can be readily effected; the flux is finely pulverised, and placed on a sheet of paper; a magnet is then drawn gently over its surface, which method of procedure will ensure the separation of all metallic particles. The formation of such globules is prevented, to a great extent, by the thin layer of fluor-spar. If the weight of metal obtained be now deducted from the weight obtained in the first weighing, the difference will be the weight of flux.

It sometimes happens that the assay is not fused, or but imperfectly so. This can happen from two causes; firstly, because the heat has not been sufficiently strong or long continued; secondly, the flux has not been employed in proper proportions, or has not been calculated to form fusible compounds with the foreign matters mixed with the oxide of iron. In the imperfectly fused buttons the iron is disseminated in globules through the whole mass of slag, or forms a scoriform button mixed with much slag, without the possibility of complete separation.

The slag ought to be well fused, colourless, transparent, or white, light-grey.

Good buttons of metal, when wrapped in pieces of thin tin plate, and struck with a heavy hammer on the anvil, flatten slightly before they break; they ought to be grey or greyish-white, and the grain fine, or tolerably fine. Bad buttons break readily without changing form, some even pulverise; they are generally very white and crystalline on the surface.

Several assays (at least two) ought to be made of the same ore, and the buttons of iron obtained should not vary, or only very little—not more than ⅛th of a per cent. The buttons always contain foreign substances (2—6 per cent.), chiefly carbon, and silicon, frequently also phosphorus, sulphur, arsenic, manganese, titanium, and chromium.

Berthier recommends the following method for estimating the other, chiefly slag-forming, components of iron-ores. The operations of this method are comprised in roasting, or calcining, to drive off any volatile or combustible matters, and in treating the ore with certain acids, the object of which is to ascertain the amount of insoluble matter, by difference of weight, before and after the action has taken place.

The hydrated ores are calcined to estimate water; and those containing manganese, to reduce it to a fixed and known state of oxidation (sesquioxide). The carbonates are roasted to expel carbonic acid, and the ores from the coal formations to burn the combustible matter with which they are mixed.

Slags and dross are also roasted to free them from charcoal. A simple calcination sometimes is sufficient, as in the case of carbonates; but where mixtures of per- and protoxide of iron are to be assayed, they must be subjected to a long roasting, in order to convert all the contained protoxide into peroxide.

Diluted and cold nitric or acetic acids are employed for minerals whose matrix is purely calcareous or magnesian, as these acids dissolve the earthy carbonates, without attacking either stones, clay, or the oxides of iron. The residue is to be well washed, dried, and weighed, and the amount of carbonates calculated by the difference. It is now to be treated with boiling hydrochloric acid, or, what is preferable, *aqua regia*. The ores which contain substances insoluble in these acids are generally of a clayey or flinty nature. These are to be weighed, and according to their weight that of the flux to be added in the assay is determined, as will be shown hereafter.

It must be borne in mind, however, that the clays are not absolutely insoluble in hydrochloric acid, for a certain quantity of alumina is always dissolved, which is generally greater in proportion to the amount present in the iron ore.

The ores containing titanium are boiled with concentrated sulphuric acid, after they have been reduced to the finest possible state of division. All the oxides of iron, titanium, and manganese, are dissolved, and the stony gangues which resist the action of this acid can be estimated. The utility of this estimation will be pointed out as we proceed.

When all the operations necessary for each particular case have been completed, we know the proportion of volatile substances, of substances soluble in acetic acid, and those insoluble in hydrochloric and sulphuric acids, contained in the substance under assay.

Let A be the weight of the rough or non-calcined ore; B the weight of the same calcined; C the weight of the fluxes in a rough state; D the weight of the same calcined; P the weight of matter insoluble in hydrochloric or sulphuric acids; R the weight of the fixed substances soluble in acetic or nitric acids,—a weight which can be readily calculated when we know the loss which the ore, not treated by acids, suffers by calcination, and the residue of the treatment of this substance by acetic or nitric acid; M the weight of the button of metal and scattered globules; S the weight of the slag; and O the loss of weight in the assay which represents the quantity of oxygen disengaged during the reduction.

The following is the disposition of the data from which, at one view, all the useful results of the assay can be determined.

In the assay has been employed:—

| | |
|---|---|
| A, rough ore = calcined ore | B |
| B, of rough fluxes added = fixed flux | D |
| Total of fixed matter | B + D |

The result has been :—

| | |
|---|---|
| Metal — M<br>Slag — S } Total | M + S |
| Loss | O |

| | |
|---|---|
| Fluxes | D |
| Vitrifiable matters | S — D |
| Substances insoluble in hydrochloric acid, &c. | T |
| Substances soluble in hydrochloric acid, &c. | S—D—T |
| Substances soluble in acetic acid | R |
| Substances insoluble in acetic acid, and soluble in hydrochloric acid | S—D—T—R |

When the iron in the substance assayed is in a known degree of oxidation, and when but little manganese is present, the quantity of oxygen O ought to correspond very nearly with the quantity of metal M produced; if it does, the assay must be correct.

A rigorous correspondence between the two numbers, however, cannot always be obtained, because the iron is not pure, but always contains carbon, so that in ordinary assays the peroxide of iron loses but from twenty-eight to twenty-nine per cent. of oxygen.

On the other hand, the quantity of iron remaining in the slag makes up in part for the carbon combined with the metal reduced; but when the assay has been made with a suitable flux, the quantity of oxide remaining is very small, and never exceeds one per cent. of the weight of the slag. When the iron is in an unknown degree of oxidation, the loss O produced in the assay gives the degree, if it has been made without accident; but if there is any doubt, and the result is of importance, the assay must be recommenced for verification. If the ferruginous matter contain manganese, and if that metal be in the state of protoxide, the verification just described can be made without modification, because the manganese dissolved in the slag is always at the *minimum* of oxidation: and when a sufficient quantity of flux is employed, the amount reduced is of no consequence. But when the manganese is in the state of red oxide, it parts with a certain quantity of oxygen on being reduced to the *minimum* of oxidation, and which quantity is estimated in the loss O, so that a perfectly accurate verification cannot be made. Nevertheless, the difference between the loss O, and the quantity of oxygen calculated from the metal M, cannot be very great, because the red oxide of manganese loses but ·068 of oxygen in its transformation to protoxide.

If the assay has been made with care, the loss of oxygen indicates the amount of iron in a very approximate manner, and nearly always with an exactitude which is surprising to those not accustomed to this kind of operation.

Titanic acid behaves in iron assays exactly as the oxides of manganese; it disengages at most but ·06 of oxygen when dissolved in the earthy glasses in contact with charcoal.

The following process of Professor Abel for the complete assay of iron and iron ores is of great interest, and therefore, may find here a proper place. It is extracted from the 'Report of Experiments on British Irons, Ores, and for the Manufacture of Cast-iron Ordnance.'

'In conducting a large series of analytical examinations of a quantitative character, which have been undertaken, not simply to furnish results of scientific interest, but with a view to be of use in considerations of a practical character, it is not only admissible, but necessary, that some discrimination should be exercised in determining the extent of detail of such examinations, so that, on the one hand, a proper knowledge be obtained of the proportions of all the important and characteristic components of a substance, while, on the other hand, no great unnecessary expenditure of time and labour be incurred in researches on the existence or proportions of constituents which occur in minute quantities in the substances examined, and have not yet been discovered to exert any influence on its chemical or physical character.

'Thus, the existence in cast iron of metals, such as titanium, calcium, magnesium, nickel, copper, lead, &c., has been traced by minute analyses of various samples by different chemical authorities, and the proportions in which they occur have also frequently been determined; but it is only when some foreign metal, such as copper, lead, or arsenic, has been found to exist in a sample of cast iron, in a larger proportion than usual, that it has been proved to exert some marked and well-established influence over the character of the metal.

'It may, therefore, be safely inferred, in the present state of our knowledge, that modifications in the properties of cast iron are not dependent upon the presence or absence of minute proportions of such foreign metals, and that researches having in view the discovery or the determination of the proportions of these constituents, do not at present possess any practical interest, and are only advisable :

'1. In conducting analyses of specimens of peculiar interest for purposes of scientific record.

'2. In undertaking special researches with a view to ascertain whether, and to what extent, these constituents influence the properties of cast iron.

'Such researches as those last referred to are unquestionably of great interest, and, if pursued to a sufficient extent, may possibly lead hereafter to important practical results. Their objects differ, however, from those of the investigation the results of which are embodied in this Report, and which was undertaken with a view to ascertain, by an application of our present knowledge of the chemical and physical properties of cast iron, the relative qualities of various descriptions of pig iron submitted by numerous manufacturers.

'There is no question, however, that it is in most instances very important to perform complete analyses of materials which are to be employed for the extraction of metals: for as these, at the high temperature necessary in the process of their reduction from ores, are endowed with powerful affinities for many of the frequent constituents of such ores, their properties will suffer more or less important modification by union with some of these components in the course of their reduction.

'The quality of the product may therefore be, to some extent (when the treatment is under control), predicted from the results of a complete examination of the materials employed. It is, consequently, not only essential to ascertain the general composition of an ore, for the purpose of determining the nature and proportions of the auxiliary materials (fluxes) to be associated with it in the reduction process, but it is also of considerable importance to ascertain whether, and in what proportions, such constituents (as foreign metals, sulphur, and phosphorus) are contained in the ores, as are likely to enter into the composition of the product and exert an influence upon its properties.

'In the chemical examination of the large series of iron samples and materials employed in their manufacture the system of analysis adopted has been based upon the principles embodied in the foregoing remarks.

'The samples of ores received from the manufacturers have been all submitted to complete analysis, excepting in the following instances:—

'1. In one or two cases where the samples were found to be identical in character with some of the series of ores recently submitted to complete analysis at the Government School of Mines, under Dr. Percy, the results of which have been published in the "Memoirs of the Geological Survey of Great Britain."

'The close resemblance of the samples to those above alluded to was established by a comparison with the descriptions given of the ores in the Government Report, by a careful examination of them made by Mr. Spiller, who performed a large number of the analyses alluded to in that Report while at the School of Mines, and was well acquainted with all the specimens examined; and lastly, by a determination of the most important constituents of the samples received (*e.g.* the oxide of iron and phosphoric acid), and a comparison of the results with those of the published analyses.

'In these instances, as a complete analysis of the samples would have involved a very considerable unnecessary expenditure of time, it was not undertaken; but the detailed analyses of the ores, to which the samples corresponded, have been extracted from the Government Report.

'2. A few of the ores received had been submitted to the roasting process. As the effect of this treatment of an ore is greatly to modify its original composition, partially expelling certain constituents, and altering the arrangement and state of combination of others, a detailed analysis would afford no correct indication of the original nature of the ore. In such instances, therefore, it was only considered important to determine the per-centages of iron and of those constituents which might affect the quality of the metal obtained from the ore—the phosphoric acid and sulphur.

'3. The ores sent by some manufacturers were identical in their nature with those from other works, and of which complete analyses have been made. In these instances the most important constituents of the samples in question have alone been determined, references having been made, when needful, to the full analyses.

'The minerals employed as fluxes were all submitted to complete analysis, except in one or two instances, when their identity with samples already analysed was established by the determination of one or two constituents.

'The examination of the fuel was partial, the only important object being to determine to what extent it might contain mineral matters possibly prejudicial to the quality of the metal reduced by its means. The examinations therefore included determinations of the amount of sulphur in the coal or coke, and the amount and character of the ash furnished by incineration. It was also considered interesting to determine the amount of coke furnished by the different samples of coal.

'The analyses of the samples of iron were, for the reasons already stated, limited in most instances to the determination of the proportions of those constituents which have already been satisfactorily proved to exert some influence on the quality of the pig iron, or on the results obtained by submitting it to subsequent processes of manufacture.

'The constituents in question are manganese, silicon, phosphorus, sulphur, and carbon.

'With reference to the last named substance, it may be necessary to observe, that almost all the specimens of pig iron examined which are included in this report were varieties of grey iron, and that but very few of the samples contained any appreciable amount of carbon in the combined form.

'It was, therefore, of no importance or interest to determine the minute portions of carbon existing in the samples in the latter form, and this was proved by special examinations of a few of the light grey samples of the series, in which the amount of combined carbon was not found to exceed at highest 0·35 per cent.: and also by the determination of the total amount of carbon in a sample of No. 1 pig iron, the result obtained being identical with that afforded by the direct estimation of the carbon existing as graphite.

'In two or three instances it was considered interesting to examine specially for certain foreign metals which had been found to exist in appreciable quantities in some of the ores from which the samples thus examined had been obtained.

## I. *Analyses of the Iron Samples.*

'*Preparation of the Sample.*—Preparatory to its examination, the metal was reduced to a suitable state of division by boring, turning, or planing. In the case of white iron it was broken to a coarse powder in a steel-crushing mortar. It was considered preferable to prepare an average sample of the pig by boring across it, so that a fair proportion of the graphite, which was occasionally concentrated towards the centre of the pig, might be included in the sample. The fine borings obtained in this way were further reduced when necessary, and thoroughly mixed by trituration in a Wedgwood mortar.

'*Chemical Analysis.*—In the analysis of pig iron the proportion of the following constituents were usually determined: manganese, carbon, silicon, sulphur, phosphorus, and, in certain cases, metals such as arsenic, lead, and copper, when their existence in appreciable quantity had been discovered in the ores from which the iron had been obtained.

'For this purpose four portions were usually weighed out:—

*a.* 100 grains, for sulphur, carbon existing as graphite, silicon, and manganese.

*b.* 50 grains, for phosphorus.

*c.* 50 to 100 grains, for determining the existence and amount of combined carbon.

*d.* 500 grains, for metals existing in the iron in minute proportions.

'**Sulphur.**—100 grains of the iron borings were slowly dissolved in concentrated hydrochloric acid, the evolved gas being passed through a solution of acetate of lead, slightly acidified with acetic acid, the sulphuretted hydrogen, disengaged together with hydrogen, precipitated the sulphide of lead, which was collected on a filter, washed, burnt, and subsequently (in the customary manner) converted into sulphate of lead, from the weight of which the per-centage of sulphur was calculated.

'The contents of the flask, after the metal had been fully acted upon, were transferred to a porcelain basin and evaporated to dryness, the mass digested with concentrated hydrochloric acid, and water afterwards added. The insoluble residue consisting of silicic acid and graphite, was collected on a filter, the filtrate being reserved for the estimation of manganese.

'**Carbon, as Graphite.**—The mixed silicic acid and graphite were separated by the action of a warm solution of pure potassa, when the silicic acid was dissolved, the graphite which remained insoluble was again collected, washed with dilute hydrochloric acid and water, and dried. It was afterwards carefully removed from the paper by scraping with a knife blade, and transferred to a platinum crucible, in which, after exposure for some time to about 300° F., it was weighed. Upon subsequently burning the graphite in a muffle, it usually left a very small quantity of reddish ash, which was deducted from the former weight.

'**Silicon.**—The amount of silicic acid, dissolved by the potassa, was recovered in the usual manner, by evaporation with hydrochloric acid ; the residue was digested with water collected, washed, dried, and weighed. The amount of the silicon in the iron was calculated from the silicic acid obtained.

'**Manganese.**—The hydrochloric acid solution, separated from the silicic acid and graphite, was divided into two equal portions, one of which, representing 50 grains of iron, was always sufficient for the estimation of the manganese. The iron in the liquid having been per-oxidised by boiling the hydrochloric acid solution, and adding occasionally a little chlorate of potassa, the acid was to a great extent neutralised by addition of carbonate of ammonia. Sufficient acetate of ammonia was afterwards added for the conversion of the chloride of iron into acetate, and the liquid was boiled, when the iron was completely separated as insoluble basic acetate. The filtrate containing the manganese was rendered alkaline with ammonia, and, after the addition of a few drops of bromine, set aside for about eighteen hours. The hydrated binoxide of manganese which had separated from the liquid, was afterwards collected, washed, dried, and ignited at a high temperature, when it was weighed as manganoso-manganic oxide ($Mn_3O_4$), which furnished, by calculation, the quantity of manganese.

'**Phosphorus.**—For the estimation of phosphorus, 50 grains of the iron borings were acted upon with warm nitro-hydrochloric acid, in a flask with a long neck, and, after complete solution of the metal, the contents of the flask were transferred to a porcelain basin and evaporated to dryness; the residue was moistened with concentrated hydrochloric acid, and again evaporated, so as thoroughly to expel nitric acid. The residue then obtained was dissolved in hydrochloric acid, the solution diluted, filtered, nearly neutralised with carbonate of ammonia, and the iron in solution reduced to protoxide by the addition of sulphite of ammonia to the gently heated liquid, and the subsequent careful addition of dilute sulphuric acid to expel excess of sulphurous acid. Acetate of ammonia and a few drops of solution of sesquichloride of iron were then added, and the liquid boiled, when the phosphoric acid was precipitated as basic phosphate of sesquioxide of iron, with some basic acetate. The liquid was rapidly filtered, with as little exposure to the air as possible, the precipitate was slightly washed and dissolved in hydrochloric acid, the solution neutralised with carbonate of ammonia, and a mixture of ammonia and sulphide of ammonia added; it was then gently heated, to ensure the conversion of the phosphate into sulphide of iron. The latter was afterwards removed by filtration, washed with dilute sulphide of ammonium, and the phosphoric acid was precipitated from the solution in the usual manner as ammonio-phosphate of magnesia, and weighed as pyro-phosphate of magnesia, from the amount of which the phosphorus was calculated.

'**Combined Carbon.**—After numerous comparative trials of the several methods in common use for determining the total amount of carbon in cast iron, that which was ultimately adopted (after necessary experiments had fully established its accuracy) consisted in dissolving the metal in an acid solution of chloride of copper, collecting and washing the insoluble residue which remained after the complete action of this solvent, and submitting it, when dry, to combustion with oxide of copper in a current of oxygen, the source of heat employed being the gas combustion furnace. The total amount of carbon in the iron was then calculated from the weight of carbonic acid absorbed by solution of potassa in the usual manner. The carbon, existing in a state of combination with the iron, was represented by the excess which this process afforded over that of the direct estimation of the carbon as graphite, in the manner already described.

'**Minute Proportions of Foreign Metals.**—About 400 or 500 grains of the iron were employed in the examination for metals precipitated by sulphuretted hydrogen, *e.g.*, lead, copper, arsenic, &c. The iron was dissolved in hydrochloric acid, and the solution, diluted and partly neutralised with carbonate of soda, was submitted to the action of sulphuretted hydrogen. After saturation with the gas, the liquid was allowed to stand at rest for several hours, and the small quantity of sediment which had subsided was examined for metals by the ordinary analytical processes.

### II.—*Analysis of the Iron Ores.*

'The analytical processes employed for the separation of the various constituents occurring in iron ores were, in great measure, identical with those employed in the examination of metallic iron. Thus, the estimation of oxide of manganese was conducted in a precisely similar manner; and, with the exception that no process of reduction was required in the case of clay ironstones and other ores containing the iron already in a state of protoxide, the phosphoric acid was determined by the same process as that employed for the estimation of phosphorus in pig iron. The amount of metallic iron, and its condition of oxidation in the ore, were determined by Marguerite's volumetrical method, with standard solution of permanganate of potassa; while the proportions of lime and magnesia, carbonic acid, water, hygroscopic and combined, insoluble residue, and the nature of this latter, were determined by following the analytical processes invariably employed in mineral analyses of this description.

'Sulphur was estimated by fusion of the ore (or, in the case of clay ironstone of the clay only) with a mixture of pure carbonate of soda and nitre; the sulphuric acid being precipitated by chloride of barium, from the acidified solution of the fused mass, and the sulphate of baryta collected, burnt, and weighed as usual. The hydrochloric acid solution of the ironstone was examined for sulphuric acid, but it was seldom that more than a trace of sulphur was detected in that form.

### III. *Analysis of the Samples of Fluxes.*

'These materials, consisting of limestone, burnt shale, &c. were analysed by a method precisely similar to that employed in the examination of the ores. In the tabulated statement showing the composition of the limestones, the amount of carbonate of lime is, in some few instances, represented by the difference, after the whole of the other constituents had been determined. In such cases the sum-total of constituents is necessarily expressed by 100 exactly.'

## B. THE ASSAY OF IRON IN THE WET WAY.

*Fuchs' Method.*—A suitable quantity (10 grs.) of the finely pulverised iron-ore (pig-iron, &c.), is dissolved in an excess of concentrated hydrochloric acid, and the resulting protochloride of iron is changed into sesquichloride of iron, by the addition of chlorate of potash; the chlorine is then to be expelled by heating the liquid. The latter is diluted with water, and perfectly clean strips of electrotyped copper (15—20 grs.) previously exactly weighed, are put into it. These operations must be done in a glass flask, tightly closed by a cork, through which a narrow glass tube passes. The liquid is now heated to the boiling-point, and kept at this temperature till the original dark-brown or yellow colour of the liquid changes to a light-yellow green or light blue-green colour. Then all the sesquichloride of iron will be reduced to protochloride in consequence of the formation of subchloride of copper. The open part of the glass-tube is now hermetically closed, and the liquid allowed to cool. The flask is then filled with hot water, and the liquid decanted from the undissolved copper. The latter is washed, first with diluted hydrochloric acid, and afterwards repeatedly with water; it is then dried and weighed. The amount of iron contained in the assayed substance may be calculated by the loss of copper, that amount of iron standing in the same proportion to the dissolved quantity of copper as the equivalent of iron (28) to the equivalent of copper (31·7). 28 parts of iron correspond to 36 parts of protoxide of iron and to 40 parts peroxide of iron.

Some iron-ores are very difficultly or only incompletely dissolved in hydrochloric acid; they must then be previously fused with carbonate of soda and potash.

The above method is based upon the fact that chemically pure hydrochloric acid is not able to dissolve copper without the presence of atmospheric air, and that this acid dissolves copper if peroxide of iron is present. The quantity of copper dissolved is then proportional to the amount of peroxide of iron present. In this process protochloride of iron and subchloride of copper are formed, $Fe_2Cl_3 + 2\ Cu = 2FeCl + Cu_2Cl$. Two equivalents of copper (63,4 parts) therefore reduce 1 equivalent of peroxide of iron (80 parts); the latter containing 2 equivalents of iron (56 parts).

In case the iron ore contains arsenic, the latter must be previously removed by fusing the iron ore with 3 parts dried carbonate of soda, and by lixiviating the fused mass.

If the iron ore contains titanium it will be necessary to decompose the chloride of iron by copper at a lower temperature than the boiling-point.

If the iron ore contains peroxide of iron together with protoxide of iron, the same process may be used to determine the quantities of both combinations. The process is then to be performed twice, once before oxidation and the second time after it. The first operation will show the quantity of peroxide of iron which the iron-ore originally contained, as the copper only acts upon peroxide of iron, and not upon protoxide of iron.

*Marguerite's Process.*—This method of analysis is based on the reciprocal action of salts of the protoxide of iron and mineral chameleon (permanganate of potash—$KO, Mn_2O_7$), whereby a quantity of the mineral chameleon is decomposed exactly proportionate to the quantity of iron.

Thus, in any given solution of iron at its maximum of oxidation, such as it more commonly exists in the mineral it is only necessary to bring it to the minimum of oxidation, and then to add gradually a solution of permanganate of potash of a known strength. As long as a trace of protoxide remains to be peroxidised the colour of the chameleon is destroyed; but it is at length noticed that the colour of the last drop added is no longer destroyed, but communicates a pink tint to the whole of the solution. This reaction indicates that the operation is terminated, and the quantity of iron in solution corresponds to the amount of permanganate added.

This reaction may be expressed by the following equation :—

$$KO,Mn_2O_7 + 10FeCl + 8HCl = 2MnCl + KCl + 5Fe_2Cl_3 8HO.$$

It will be seen that 1 equivalent of permanganate of potash is capable of peroxidising 10 equivalents of protoxide of iron. It is hardly necessary to mention that the solution of the iron should contain a sufficient excess of acid to hold in solution the peroxide of iron formed, and also the protoxide of manganese and potash resulting from the decomposition of the permanganate.

If now we consider the various operations in the process, we shall find they consist of the following :—

1. Dissolving the ore in an acid ; hydrochloric acid, for example.

2. Treating the solution of the persalt of iron which results by sulphite of soda, to reduce it to the state of protosalt, and to boil it in order to expel the excess of sulphurous acid.*

3. Adding afterwards with precaution the solution of permanganate of potash until the pink tint appears, and then reading off on the graduated tube the number of divisions used.

Now it will be perceived there are two conditions to fulfil ; the first, to effect a complete reduction, for the persalts of iron do not react on the chameleon,—all that remained at the maximum of oxidation will escape the action of the chameleon, and consequently will not be estimated as iron; the second, to expel by ebullition the whole of the sulphurous acid in excess, which, in contact with the permanganate, would take from it the oxygen necessary to form sulphuric acid, and thus react in the same manner as the iron. But it is easily demonstrated by experiment, that the solution of a persalt of iron, treated with a sufficient quantity of sulphite of soda, is on the one hand completely reduced to its minimum of oxidation, and on the other hand does not contain the most minute trace of sulphurous acid after a few minutes' ebullition.

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* As it is important to employ a sufficient quantity of the sulphite of soda to render the reduction of the persalt of iron to the state of protosalt complete, and yet to leave sufficient hydrochloric acid in excess in the solution, it is advantageous to use a definite and known quantity. For this purpose 4 oz. of crystallised sulphite of soda are dissolved in a quart of water, and a pipette which contains 2 oz. is used to measure the quantity added to each assay. 87½ grs., which are contained in the 2 oz. of the pipette, are more than sufficient to reduce 20 grs. of iron ; but this excess is necessary to ensure the entire reduction of the persalt to protosalt. Metallic zinc is frequently used for this purpose, and, according to Reynolds (*Chemical News*, November 5, 1864), sulphuretted hydrogen is preferable to both. Dr. Percy also recommends zinc.

A question here naturally presents itself, whether the salts of iron, reduced to their minimum, do not absorb oxygen again with great rapidity, and thus exert an influence on the results of the analysis: the following experiment, however, will remove all doubts on this head:—At this stage of the operation the solution was exposed to the contact of air for four hours, and the test-liquor then added; a quantity of this was required exactly equal to that which was necessary when the analysis was prosecuted without any delay. This fact proves that the protosalts of iron in an acid solution are converted into persalts very slowly.

It becomes important to ascertain whether, in the ores of iron there may not exist substances capable of reacting on the chameleon and thus rendering the estimation of the metal erroneous.

On examining the composition of the greater number of the ores described by various authors, and particularly by MM. Berthier and Karsten, we find that they are most ordinarily composed of the following substances:—

| Ores. | | Metals. |
|---|---|---|
| Iron. | Phosphoric acid. | Cobalt. |
| Manganese. | Lime. | Nickel. |
| Zinc. | Alumina. | Titanium. |
| Arsenic. | Magnesia. | Chromium. |
| Copper. | Silica. | Tungsten. |

The presence of zinc, manganese, titanium, tungsten, phosphoric acid, lime, magnesia, alumina, and silica, do not at all interfere with the accuracy of the results. Cobalt, nickel, and chromium, notwithstanding the peculiar colour of their solutions, do not in the least prevent the appreciation of the peculiar rose-pink tint of the mineral chameleon.

Arsenic and copper, then, are the only substances among those capable of producing a discrepancy in the analysis; as, under the influence of the sulphurous acid, the arsenic acid becomes arsenious acid, and the salts of peroxide of copper become salts of the protoxide, and afterwards withdraw oxygen from the permanganate of potash.

It is true that the ores containing arsenic are of little importance in a commercial point of view, for the iron produced from them is of so inferior quality as to be generally rejected; nevertheless, it has been considered right to give the method of analysis in cases where it occurs, and a slight modification of the general process is sufficient.

The operation is carried on as usual, except that, after having boiled the solution to expel the excess of sulphurous acid, a piece of pure laminated zinc is added, which, acting upon the hydrochloric acid, disengages hydrogen; arsenic and copper are thereby reduced and precipitated in the metallic state. When the solution of the zinc is complete, the liquid is filtered from the precipitated particles of arsenic and copper, which would otherwise be re-oxidised; and, after washing the filter three or four times with common water, the addition of the normal test-liquor is proceeded with.

The method of preparing the permanganate of potash test-liquor has already been given in the chapter on Volumetric Analysis.

Permanganate of potash is a preparation of great stability, and may be preserved for a very long time without undergoing any alteration, provided it be defended from the contact of organic matters and kept in a glass-stoppered bottle. To convert its solution into a test-liquor of known value, 20 grs. of pure iron, such as pianoforte wire, are dissolved in about 1 oz. of strong hydrochloric acid, free from iron; after the disengagement of the hydrogen has ceased, and the solution is complete, the liquid is diluted with about 1 pint of water.*

The solution of permanganate of potash is then added, drop by drop, until a slight pink colour is manifest, and the number of divisions on the tube necessary to produce this effect carefully noted; this number is then employed to reduce into weight the result of an analysis of an ore.

When the solution of chameleon is too concentrated, it is easy, by adding the proper quantity of water, to reduce it to one-half, one-fourth, or one-fifth, so that 2 oz. shall be as nearly as possible equivalent to 20 grs. of iron.

Fresenius† makes the following observations on the determination of iron in hydrochloric acid solution by the foregoing method. This process was long considered to be the most convenient and best for the estimation of iron. But its glory is now departed, since Löwenthal and Lenssen have shown that in solutions containing hydrochloric acid, it is essential that the standardising of the reagent and the actual analysis be performed under the same circumstances as regards dilution, amount of acid, and temperature. Besides the proper reaction, $10FeO + Mn_2O_7 = 5Fe_2O_3$ $2Mn\ O$, the collateral reaction $7HCl + Mn_2O_7 = 5Cl + 2MnCl + 7HO$ also takes place, in consequence of which a little chlorine is liberated. This chlorine does not oxidise the protoxide of iron in the case of considerable dilution, but there occurs a condition of equilibrium in the fluid containing protoxide of iron, chlorine, and hydrochloric acid, which is destroyed by addition of a further quantity of either body (Löwenthal and Lenssen). But since it is difficult to preserve the above condition of obtaining correct results, the following proceeding is adopted.

'Standardise the permanganate by means of iron dissolved in dilute sulphuric acid, make the iron solution to be tested up to ¼ litre, add 50 c.c. to a large quantity of water acidified with sulphuric acid, add permanganate from burette, then again 50 c.c. of the iron solution, permanganate again, &c.

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* It is necessary to use solutions very dilute and cold, in order to prevent the hydrochloric acid in excess from reacting on the chameleon and disengaging chlorine.

† Fresenius's *Quantitative Analysis*, 4th edition, p. 191.

&c. The numbers obtained at the third and fourth time are taken. These are constant, while that obtained the first time, and sometimes also the second time, differs. The result multiplied by 5 gives exactly the quantity of permanganate proportional to the amount of protoxide of iron present.

'I believe that the reason why the attention of analysts was not previously directed to the important influence of hydrochloric acid in this process, lay in the fact that it was not customary to crystallise the permanganate before employing it—the crude solution, which contains much chloride of potassium, being used. The experiments were consequently performed in the presence of free hydrochloric acid, even when sulphuric acid alone was employed for dissolving or acidifying. Hence the differences between the results with sulphuric and hydrochloric acid solutions were not so large as they are now, when we work with the pure permanganate.'

In reference to this process, Mr. J. P. Blunt has communicated the following observations to the 'Chemical News.' 'In the course of some late experiments on the estimation of the value of iron ore by means of permanganate of potash, I met with much annoyance from the inconstancy of the results obtained. The reducing agent used was metallic zinc, and the ore, being a very rich protosesquioxide containing nearly 65 per cent. of iron, required a large quantity of hydrochloric acid for its decomposition, and a corresponding amount of zinc for its reduction. My first suspicions fell upon the zinc, and I accordingly dissolved a little of it in hydrochloric acid, and, when the solution was quite complete, added a few drops of the permanganate solution; the colour immediately disappeared, and a strong and unmistakeable odour of chlorine was evolved. This led me to conclude that my failures had arisen from an insufficient dilution of the hydrochloric acid used, and I determined to institute experiments with the object of discovering what amount of dilution was necessary in order to prevent the decomposition of permanganate of potash by hydrochloric acid. The results of these experiments I now submit, as I believe that it is not generally known that any but the strongest solutions of hydrochloric acid have such an effect, and a knowledge of this may preserve those engaged in similar inquiries from the vexation and perplexity to which I have been subjected.

'Nine solutions of hydrochloric acid were prepared of successive degrees of dilution. No. 1, containing 1 part of hydrochloric acid, of 1·45 specific gravity, to 1 part of water. No. 9, containing 1 part of the same acid to 9 of water. Three or four drops of the permanganate solution were mixed with each; the results were as follows:—

'No. 1. The colour disappeared immediately.

'No. 2. The colour disappeared in one or two seconds.

'No. 3. The colour disappeared in about half a minute.

'No. 4. The colour disappeared in one minute and a half.

'No. 5. The colour disappeared in about six minutes.

'No. 6. Just coloured after nine minutes.

'No. 7. Retains its colour for nearly a quarter of an hour, but smells strongly of chlorine after about a minute.

'No. 8. Smells of chlorine after about two minutes.

'No. 9. Smells very faintly after three minutes.

'In inspecting these results it should not be forgotten that a dilute solution of permanganic acid, such as that formed in the experiments, decomposes spontaneously in a short time, and this presents oxygen in a nascent state eminently fitted for the decomposition of hydrochloric acid; it is probable that the smell of chlorine in experiments No. 6, 7, 8, and 9, may be partially attributed to this, but there would be great danger of the faint colour—which is sufficient to the practised eye to show the completion of the process of estimation—being destroyed by such a solution as No. 5, and as drop after drop was added, the same action would continue, and would seriously vitiate the results.'

*Dr. Penny's Process.*—The following method of determining the amount of iron in a sample by means of a normal solution, has been contrived by Dr. F. Penny, who was led to substitute bichromate of potash for permanganate of potash, as recommended by Marguerite, and just described. The reason of employing the bichromate is, that it is an unchangeable salt, whilst the permanganate sometimes undergoes decomposition, so that its strength is variable, and each series of experiments made with it requires a separate verification by means of a weighed quantity of pure iron. This inconvenience is avoided in Dr. Penny's method, which is described in his own words as under:—

‘In the first series of experiments, pure harpsichord wire was dissolved with every care in hydrochloric acid, and bichromate of potash, added to the solution until the conversion of the protochloride of iron into the perchloride was complete. I obtained the following results:

| | | Iron. | | | Bichromate. | |
|---|---|---|---|---|---|---|
| ‘Exp. | I. | 50 | grains | required | 44·4 | grains. |
| „ | II. | 39·7 | „ | „ | 35·2 | „ |
| „ | III. | 48·3 | „ | „ | 42·8 | „ |
| „ | IV. | 55·3 | „ | „ | 49·2 | „ |

‘The mean of these results is, 100 parts of iron to 88·75 of bichromate.

‘In the second series of experiments protosulphate of iron was employed. This salt was made from protosulphide of iron, and purified most carefully by repeated crystallisation. A known quantity of it was dissolved in water, acidulated with either pure hydrochloric or sulphuric acid, and the solution treated with bichromate:—

| | | Sulphate of Iron. | | | Bichromate. | |
|---|---|---|---|---|---|---|
| ‘Exp. | I. | 100 | grains | required | 17·90 | grains. |
| „ | II. | 180 | „ | „ | 32·10 | „ |
| „ | III. | 150 | „ | „ | 26·82 | „ |
| „ | IV. | 120 | „ | „ | 21·40 | „ |

‘These experiments give the ratio of 100 parts of sulphate of iron to 17·867 of bichromate, or 100 of iron to 88·71, which corresponds very closely to the mean result obtained with the metallic iron. Moreover, I performed a series of similar experiments with the neutral chromate of potash, and obtained results completely confirmatory of the general accuracy of the foregoing experiments. We may, therefore, I think, safely conclude that 100 parts of metallic iron correspond to 88·75 of the bichromate of potash, and that 100 of the latter are equal to 112·67 of the former.

‘I shall now proceed to describe the method of employing the bichromate of potash for the determination of the amount of iron in clay-band and black-band ironstone. I shall be purposely minute, as I particularly desire that the process may be serviceable to those who, from their pursuits in life, are interested in the value and quality of ironstone, and who may be imperfectly acquainted with analytical operations.

'A convenient quantity of the specimen is reduced to coarse powder, and one-half at least of this still further pulverised, until it is no longer gritty between the fingers. The test solution of bichromate of potash is next prepared. 44·4 grs. of the salt in fine powder are weighed out, and put into an alkalimeter (graduated into 100 equal divisions), and tepid distilled water afterwards poured in until the instrument is filled to 0. The palm of the hand is then securely placed on the top, and the contents agitated by repeatedly inverting the instrument, until the salt is dissolved and the solution rendered of uniform density throughout. It is obvious that each division of the solution thus prepared contains 0·444 gr. of bichromate, which corresponds to ½ a grain of metallic iron. The bichromate of potash used for this process must of course be purchased pure, or made so by repeated crystallisation, and it should be thoroughly dried by being heated to incipient fusion.

'100 grs. of the pulverised ironstone are now introduced into a Florence flask, with 1½ oz. by measure of strong hydrochloric acid, and ½ an ounce of distilled water. Heat is cautiously applied, and the mixture occasionally agitated, until the effervescence caused by the escape of the carbonic acid ceases; the heat is then increased, and the mixture made to boil, and kept at moderate ebullition for ten minutes or a quarter of an hour. During these operations it will be advisable to incline the flask, in order to avoid the projection, and consequent loss, of any portion of the liquid by spirting. About 6 oz. of water are next added, and mixed with the contents of the flask, and the whole rapidly transferred to an evaporating basin. The flask is rinsed several times with water, to remove all adhering solution.

'Several small portions of a weak solution of pure red prussiate of potash (containing one part of the salt to 40 of water) are now dropped upon a white porcelain slab, which is conveniently placed for testing the solution in the basin during the next operation.

'The prepared solution of bichromate of potash in the alkalimeter is then added very cautiously to the solution of iron, which must be repeatedly stirred, and as soon as it assumes a dark greenish shade, it should be occasionally tested with the red prussiate of potash. This may be easily done by taking out a small quantity on the top of a glass rod, and mixing it with a drop of the solution on a porcelain slab. When it is noticed that the last drop communicates a distinct red tinge, the operation is terminated. The alkalimeter is allowed to drain for a few minutes, and the number of divisions in the test-liquor consumed read off. This number multiplied by two gives the amount of iron per cent. in the specimen of iron-stone, assuming that, as directed, 100 grs. have been used for the experiment. The necessary calculation for ascertaining the corresponding quantity of protoxide is obvious.

'When black-band ironstone is the subject of analysis, or when the ore affords indications, by its appearance or during the treatment with hydrochloric acid, that it contains an appreciable quantity of carbonaceous matter, then the hydrochloric acid solution must be filtered before being transferred to the basin, and the filter, with the insoluble ingredients must be washed in the usual way with warm distilled water, slightly acidulated with hydrochloric acid until the filtrate ceases to give a blue colour with the red prussiate of potash. In those cases, also, where the presence of iron pyrites in the ironstone is suspected, it will be necessary to remove the insoluble matter by filtering before applying the bichromate solution; but with ironstones in which the insoluble ingredients are merely clay and silica, filtration is not essential.

'Now it is evident that the foregoing process, so far as I have described it, serves for the determination of that portion of iron only which exists in the ore in the state of protoxide. But many specimens of the common ironstone of this country contain appreciable quantities of peroxide of iron, which, being unacted upon by the bichromate of potash, would escape estimation by the present method. By an addition and easy operation, however, the amount of metallic iron in this ingredient may be likewise determined. It is only necessary to reduce it to the minimum state of oxidation and then to add the bichromate as previously directed.

'The best and most convenient agent for effecting the reduction of the persalts of iron is sulphite of soda. The only precaution to be observed is to use it in sufficient quantity, and at the same time to take care that the iron solution contains excess of acid. When the reduction is complete, a few minutes' ebullition suffices to decompose the excess of sulphite of soda, and effectually to expel every trace of sulphurous acid.

'In order to test the exactness of this mode of estimating the iron of the peroxide, I made several experiments with peroxide prepared from known quantities of pure iron wire. The peroxide was thoroughly washed, dissolved in hydrochloric acid, reduced with sulphite of soda, and after complete expulsion of the excess of sulphurous acid, the solution was diluted with water and treated with bichromate of potash. I select three of the experiments :—

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 'Exp. | I. | 10 | grains of iron | consumed | | 8·87 | of bichromate. |
| „ | II. | 18 | „ | „ | „ | 15·94 | „ |
| „ | III. | 25 | „ | „ | „ | 22·15 | „ |

'The mean of all my experiments on this point gives the ratio of 100 of iron to 88·6 of bichromate, which is in close accordance with the former results.

'Whenever, therefore, the ore of iron contains peroxide it will be necessary to add sulphite of soda to the hydrochloric acid solution before the addition of the test-liquor from the alkalimeter. The sulphite should be dissolved in distilled water, and added to the solution of iron in small successive portions, until a drop of the liquor gives merely a rose-pink colour with sulphocyanide of potassium, which indicates that the reduction of the persalt of iron is sufficiently perfect. The liquor is now heated till the odour of sulphurous acid is no longer perceptible. These operations should be performed while the solution is in the flask, and before it is filtered or transmitted to the basin.

'I will here mention for the guidance of those who may not be fully aware of the reactions of the oxides of iron, that the existence of an appreciable quantity of peroxide in the ironstone may be readily discovered by dissolving (as directed in the process) 39 or 40 grs. of the ore in hydrochloric acid, diluting with about 8 oz. of water, filtering and testing a portion of the solution with sulphocyanide of potassium. If a decided dark blood-red colour is produced, the quantity of peroxide in the stone must be determined; but if the colour is only light red or rose-pink, the proportion is exceedingly small, and for practical purposes not worth estimating. Of course, when the specimen of ironstone has an ochrey or a reddish appearance on the surface or in the fracture, the presence of a large proportion of peroxide is indicated, and its exact quantity must be determined.

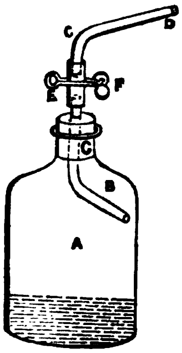
'In conclusion, I must not omit to notice one or two circumstances which appear at first to militate against the accuracy of this process. It may be questioned whether solutions of the protosalts of iron do not absorb oxygen so rapidly from the air as to influence the results obtained by this method. Marguerite has shown (see *ante*), and my own observations completely confirm his statement, that protosalts of iron, in an acid solution, become peroxidised very slowly; and, in a particular experiment, I found that contact with the air during several hours caused no diminution in the quantity of bichromate of potash required. As the process may be completed in a few minutes, it is certain that no inaccuracy can arise from this cause.

'It is also important to inquire whether the chromic acid in the chromates of potash may not be partially deoxidised by hydrochloric acid alone without the presence of a protosalt of iron. Such a reaction would obviously give rise to a serious error. It is well known that concentrated hydrochloric acid rapidly decomposes the chromic acid of the chromates when aided by the application of heat. But I have satisfied myself, by numerous experiments, that this acid exerts very little appreciable action upon dilute solutions of the chromates of potash, either cold or warm, and that the action is only partial even after continued ebullition; so that the present method is free from inaccuracy on this account.'

*M. Mittenzwey's Process.*—M. Moritz Mittenzwey has described a very good process for estimating iron by means of tannic acid. The estimation can be conveniently made in the simple apparatus here figured and described.

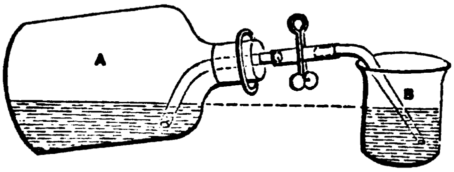
The air in a bottle, A, fig. 76, capable of holding about a litre and a half, communicates with the atmosphere by the bent tubes, B and C, the latter being drawn out at the end D to the diameter of about one or one and a half millimètres. The two glass tubes are united by means of a moderately long piece of india-rubber tubing, E, provided with a pinchcock, F, to close it; and the lower glass tube is fixed in the neck of the bottle by a bored cork, or, better, a caoutchouc stopper.

FIG. 76.



In executing the analysis it is absolutely necessary that the air in the bottle should be perfectly renewed, and the temperature of all reaching the fluid be the same as that of the laboratory. As soon as the absorbing liquid (which should amount to 150 or 250 c.c.) is prepared, the bottle should be perfectly closed, and then the pinchcock opened just for a moment, so that the pressure of the internal and external air may be equalised. The absorption of the oxygen is then hastened by strongly shaking the bottle, which must be wrapped in a cloth to avoid raising the temperature by the warmth of the hand. After each shaking, water must be

FIG. 77.



allowed to flow into the bottle, A, from a weighed quantity in a beaker, B, fig. 77, so that the fluid in the two vessels may attain the same level, as shown in the drawing. The experiment is ended when, after repeated shakings, no more water runs from B to A, and the difference in the weight of the water in the beaker in *grammes* gives the amount of oxygen absorbed in cubic centimètres, which can be corrected for the standard temperature and pressure.

In order to apply this to the estimation of iron compounds these must be reduced to the state of protoxide by means of zinc, and the excess of acid neutralised with caustic potash or soda. (Ammonia and the carbonated alkalis must be avoided.) The solution is then poured into the absorption-flask, and pieces of potash wrapped in paper are then dropped in. The absorption is complete in a very short time. For accuracy this process is second to none, and may be recommended in preference to that of Marguerite and Fuchs, since it requires fewer precautions. 50 c.c. of a solution of protoxide which contained 1·395 Fe absorbed in three experiments 148·0 c.c., 148·44 c.c., and 148·4 c.c. of oxygen at 19° C.; the mean = 148·28 c.c., which at this temperature weigh 0·1987 gramme, answering in 1·391 grammes of iron.

4. *Titration of Iron by Protochloride of Tin.*—Mr. Sutton, in his excellent 'Volumetric Analysis' before quoted, gives the following directions for the direct titration of iron by protochloride of tin.

The principle involved in this reaction is, in fact, simply a reversion of the ordinary process by permanganate and bichromate. In the case of these two reagents, the amount of oxygen *given up* by them is the measure of the quantity of iron, whereas with protochloride of tin, it is the amount *taken up* by it that answers the same purpose.

Fresenius (in his 'Zeitschrift für Analytische Chemie,' part 1, page 26) has recorded a series of experiments made on the weak points of this process, and gives it as his opinion that it is most accurate and reliable with proper care, without which, of course, no analytical process whatever is worth anything. The summary of his paper is as follows:—

*a.* A solution of peroxide of iron of known strength is first prepared, by dissolving 10·03 grm. fine pianoforte wire (=10 grm. pure iron) in pure hydrochloric acid, adding chlorate of potash to complete oxidation; boiling till the excess of chlorine is removed, and diluting the solution to 1 litre.

*b.* A clear solution of protochloride of tin, of such strength that about equal volumes of it and the iron solution are required for the complete reaction.

*c.* A solution of iodine in iodide of potassium, containing about 0·005 grm. iodine in 1 c.c. (if the operator has the ordinary decinormal iodine solution at hand, it is equally applicable.) The operations are as follows:

1. 1 or 2 c.c. of the tin solution are put into a beaker with a little starch liquor, and the iodine solution added from a burette till the blue colour occurs; the quantity is recorded.

2. 10 c.c. of the iron solution = 0·1 grm. iron, are put into a small flask with a little hydrochloric acid, and heated to gentle boiling (preferably on a hot plate), the tin solution is then allowed to flow in from a burette until the yellow colour of the solution is nearly destroyed, it is then added drop by drop, waiting after each addition until the colour is completely gone and the reduction ended. If this is carefully managed there need be no more tin solution added than is actually required; however, to guard against any error in this respect, the solution is cooled, a little starch liquor added, and the iodine solution added by drops until a permanent blue colour is obtained. As the strength of the iodine solution compared with the tin has been found in 1, the excess of tin solution corresponding to the quantity used is deducted from the original quantity, so that by this means the volume of tin solution corresponding to 0·1 grm. iron is found.

The operator is, therefore, now in a position to estimate any unknown quantity of iron which may exist, in a given solution, in the state of peroxide, by means of the solution of tin.

If the iron should exist partly or wholly in the state of protoxide, it must be oxidised by the addition of chlorate of potash, and boiling to dissipate the excess of chlorine, as described in 2.

Example : 10 c.c. of iron solution, containing 0·1 grm. iron, required 15 c.c. of tin solution.

A solution, containing an unknown quantity of iron, was then taken for analysis, which required 12 c.c., consequently, a rule of three sum gave the proportion of iron as follows :—

15 : 0·1 grm. :: 12 : 0·08 grm.

It must be remembered that the solution of tin is not permanent, consequently it must be tested every day afresh. Two conditions are necessary in order to ensure accurate results.

1st. The iron solution must be tolerably concentrated, since the end of the reduction by loss of colour is more distinct; and, further, the dilution of the liquid to any extent interferes with the quantity of tin solution necessary to effect the reduction. Fresenius found that by diluting the 10 c.c. of iron solution with 30 c.c. of distilled water, $\frac{1}{10}$ of a c.c. more was required than in the concentrated state. This is, however, always the case with protochloride of tin in acid solution, and constitutes the weak point in Streng's method of analysis by its means; it would seem that dilution either predisposed it to rapid oxidation, or that water had the power within itself to communicate a certain proportion of oxygen to it.

2nd. The addition of the tin solution to the iron must be so regulated that only a very small quantity of iodine is necessary to estimate the excess—if this is not done another source of error steps in, namely the influence which dilution, on the one hand, or the presence of great or small quantities of hydrochloric acid on the other, are known to exercise over this reaction; practically it was found that where the addition of tin, to the somewhat concentrated iron solution, was cautiously made so that the colour was just discharged, the mixture then rapidly cooled, starch added, and iodine till blue, that the estimation was as reliable as by any other method.

The following examples are from Fresenius.

The standard iron solution contained 10 grm. in the litre; 10 c.c. were therefore equal to 0·1 grm. iron. 1 c.c. tin solution required 3·62 c.c. iodine.

Exp. 1. 9·97 c.c. of the above iron solution required 11·6 c.c. tin solution and 1·23 c.c. iodine.

Exp. 2. 9·87 c.c. iron solution required 11·26 c.c. tin and 0·44 c.c. iodine. Calculated for 0·1 grm. iron, the above experiments show that—

1 = 11·294 c.c. tin solution
2 = 11·287 " "
Mean 11·2905 " "

3·8204 grm. brown hæmatite ore was heated with concentrated hydrochloric acid until decomposed, then diluted somewhat, filtered, and the solution made up to 500 c.c.

Exp. 1. 100 c.c. required 43·69 c.c. tin solution and 0·26 c.c. iodine.

Exp. 2. 100 c.c. required 44·15 c.c. tin and 2·12 c.c. iodine, therefore,—

1 = 43·62 c.c. tin solution
2 = 43·57 " "
Mean 43·60 " "

The following equation expresses the result.

11·2905 SnCl : 0·1 Fe :: 43·60 : $x$=0·3862 grm. iron in 100 c. c. or 50·54 per cent. of iron in the ore.

A determination of the iron, in the same sample of ore, by permanganate, executed with the greatest care, gave 50·58 per cent.

The tin solution is best prepared by placing fragments of pure tin at the bottom of a beaker, laying a small platinum crucible or cover upon them, and covering the whole with equal parts of pure hydrochloric acid and water: a large watch-glass or porcelain capsule should be placed on the top of the beaker, to exclude air and prevent loss by spirting.

The contact of the platinum with the tin sets up a galvanic current which materially hastens the solution of the tin without at all affecting the platinum; when the acid is all saturated, it may be poured off and fresh added until sufficient solution has been obtained. The whole, freely acidified and diluted to a convenient strength, should be placed in a well-stoppered bottle, with a few fragments of tin; its strength, which is constantly lessening to a slight extent, must be found before using it.

*Quantitative Determination of all the Constituents usually present in an Iron Ore.*—The ordinary constituents of clay ironstone (which is about the most complex, and the detail of whose analysis will be the most useful) are the per- and protoxides of iron, oxide of manganese, alumina, magnesia, lime, potash, soda, sulphur, phosphoric acid, carbonic acid, silica, and water.

Some iron ores dissolve very readily in hydrochloric acid or in *aqua regia*; others do not, even when they are in a very fine state of division; but all do readily after fusion with an alkali, or an alkaline carbonate,—as of potash or soda, hence it is advisable to fuse the finely pulverised ore with an alkali previous to attempting its solution in an acid.

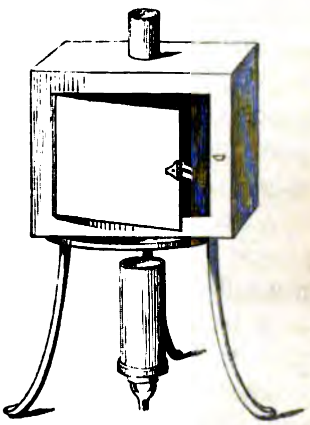
In determining the amount of iron, the author recommends Dr. Penny's process.

*Determination of Silica, Oxide of Iron, and Oxide of Manganese.*—The ore must be reduced to the finest possible state of division, a small quantity placed in a test-tube, and boiled for some time with hydrochloric acid. If it completely decomposes it need not be submitted to fusion with carbonate of soda, but 100 grains may be at once weighed off, and treated in a Florence flask with about 2 ounces of hydrochloric acid, gradually heated to ebullition, and that temperature maintained until perfect decomposition has ensued. If, on the other hand, the ore does not completely decompose, 100 grains must be carefully mixed with 500 or 600 grains of carbonate of soda placed in a platinum crucible and fused at a bright red heat; the fusion must continue about half an hour. It may be here mentioned that the platinum crucible, previous to its introduction into the furnace, must be placed in one of clay furnished with a cover, to protect it from the injurious effect of contact with the fuel.

When the platinum crucible and its contents are cold, it is placed in a large evaporating basin, and pure dilute hydrochloric acid poured over it: the fused mass dissolves with effervescence, and more acid must be gradually added as seems necessary, until no further action takes place. The solution being finished, the crucible is removed, washed with distilled water, and the whole, together with the washings, evaporated to dryness. The solution obtained in the first case, in which the ore was wholly decomposable by hydrochloric acid alone, is also to be evaporated to dryness. The object of this evaporation is the conversion of the silica the ore may contain from a partially soluble to a completely insoluble state, so that the whole of it may be collected and weighed.

Towards the end of the operation, the partially-dried mass must be continually stirred, in order to prevent losses by the spirting which will otherwise take place. When cold, the contents of the basin are moistened with hydrochloric acid, and the whole left to itself for about one hour. It is then mixed with a small quantity of distilled water, gently warmed and thrown upon a filter. Every constituent of the ore, with the exception of the silica, will pass through the filter in a liquid state. The silica remaining in the filter is to be well washed with hot water, dried,* ignited in a platinum crucible, and weighed.

Fig. 78.



To the liquid filtered from the silica and with which the washings have been incorporated add a few drops of nitric acid, and boil; when cool, add gradually pure precipitated carbonate of baryta until in excess, which point may be ascertained by cessation of effervescence, and by some of the carbonate remaining undissolved. The whole is now to be kept at a gentle heat for about an hour, and then poured on a filter, in which will remain the peroxide of iron, alumina, and phosphoric acid, together with the excess of carbonate of baryta employed. The liquid which has passed through the filter is mixed with excess of sulphide of ammonium, covered with a glass plate to exclude air, and left to itself for four or five hours. If any manganese were present in the ore, it will now be thrown down as a flesh-red precipitate, which must be collected on a filter, washed, dissolved in a small quantity of hydrochloric acid, the solution filtered, and excess of carbonate of soda added: carbonate of manganese is precipitated, which is collected on a filter, washed, dried, ignited and weighed as red oxide, every 100 parts of which correspond to 93 parts of the protoxide of manganese, in which state it usually exists in the ore. The weight so obtained gives the percentage. The mixed precipitate of oxide of iron, alumina, carbonate of baryta, and phosphoric acid remaining on the filter, is dissolved in a small quantity of hydrochloric acid, and the amount of iron ascertained by Dr. Penny's process, as already described. As the iron is in the state of peroxide, its reduction to protoxide must be effected by sulphite of soda, according to the method already given.

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* The most convenient form of apparatus for drying precipitates, filters, &c. in analysis, is a little water-oven, called a 'water-bath' (see fig. 78). It consists of a double box of copper or tin plate about six inches square, with water between the casings, which is kept in a state of ebullition by means of a gas flame or spirit lamp.

*Determination of Lime and Magnesia, and part of Phosphoric Acid.*—Dissolve another 100 grains of ore with the precautions already pointed out, only in this case the silica may be rejected, and treat the solution by the following process, which was contrived by Fresenius:—

The solution is heated to ebullition in a flask, and reduced with sulphite of soda, then precipitated with carbonate of soda, and boiled with excess of caustic soda until the precipitate appears black and granular. It is allowed to subside, the clear liquid poured off, the precipitate washed by decantation with hot water, and finally brought upon a filter of close texture and washed with hot water.

*Treatment of the Precipitate.*—The precipitate is again transferred, together with the filter, into the flask, and digested with hydrochloric acid. When no more black particles are perceptible it is filtered; the filter is left whole, a little water poured over it, and the flask inclined so that it remains hanging by the side while the liquid runs off: in this manner it may be quickly and completely washed. The filtered solution is reduced with sulphite of soda, heated to boiling, mixed with a few drops of chlorine water, then with an excess of acetate of soda; and when the liquid or precipitate has not a reddish tint, chlorine water is added until this is the case. The whole is boiled until the precipitate has separated, filtered hot, and the precipitate, consisting of phosphate and some basic acetate of the peroxide of iron, washed.

To the solution just filtered from the phosphate of iron, add ammonia and sulphide of ammonium, and filter while hot; this removes manganese and iron, leaving lime and magnesia alone in solution. The whole is filtered while hot, and the precipitate remaining on the filter rejected. To the filtered solution is added excess of solution of oxalate of ammonia: this throws down insoluble oxalate of lime, which must be collected on a filter, washed, dried, and ignited at a low red heat. The residue is now carbonate of lime, every 100 parts of which correspond to 56·29 parts of lime.

To the solution filtered from the oxalate of lime, and which contains the magnesia, add excess of phosphate of soda, agitate briskly, and set aside for twelve hours; then collect the crystalline precipitate of ammonio-phosphate of magnesia on a filter, wash it with water containing a little ammonia, dry and ignite it; weigh the resulting pyro-phosphate of magnesia: every 100 parts correspond to 36·67 parts of magnesia.

The precipitate containing the perphosphate and basic acetate of soda is dissolved in hydrochloric acid, reduced with sulphite of soda, boiled for some time with excess of caustic soda, and filtered. The filtered solution which contains the phosphoric acid is supersaturated with hydrochloric acid, and placed aside for future operation.

*Treatment of the alkaline solution poured off from the first black precipitate. Determination of Alumina and remainder of Phosphoric Acid.*—The solution is acidulated with hydrochloric acid, a little chlorate of potash added, and then boiled; it is then precipitated with ammonia (avoiding a large excess), and chloride of barium added as long as a precipitate appears. After digesting for some time it is filtered. The precipitate, which contains the whole of the alumina and phosphoric acid, is collected on a filter, washed with a little water, and dissolved in as little hydrochloric acid as possible. The solution is saturated with precipitated carbonate of baryta, gently warming; an excess of caustic soda is added, and the heat still kept up. Any baryta contained in the solution is removed by carbonate of soda, which is added until no further precipitation takes place. The whole of the alumina is now in solution, and the whole of the phosphoric acid in the precipitate.

The solution is rendered acid with a little hydrochloric acid, boiled with a small quantity of chlorate of potash, precipitated with excess of ammonia, and allowed to stand for a few hours; after which the precipitated alumina is collected on a filter, washed, dried, ignited, and weighed: its amount represents the per-centage of alumina in the ore.

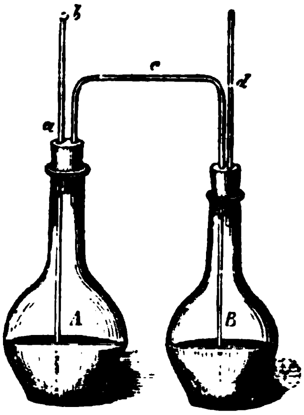
The precipitate containing the phosphoric acid is dissolved in hydrochloric acid, the baryta precipitated with dilute sulphuric acid, which is added until no further precipitate ensues; the liquid and precipitate placed in a warm situation until the former is quite bright: it is then filtered, and to the filtered liquid is added the small portion reserved, as before directed: excess of ammonia is added to the mixture, then some chloride of ammonium, and lastly sulphate of magnesia. The phosphoric acid is precipitated as the ammonio-phosphate of magnesia, which is washed, dried, and ignited, with the precautions already pointed out. Every 100 parts correspond to 63·33 parts of phosphoric acid.

*Determination of Potash and Soda.*—If the ore be completely decomposible by hydrochloric acid, dissolve at once 100 grains in that liquid; if not, fuse the same quantity with four times its weight of hydrate of baryta in a platinum crucible: treat with hydrochloric acid, and separate the silica precisely as already described. To the filtered solution add an excess of baryta water; this precipitates everything but the potash and soda and part of the lime. Throw the whole on a filter, well wash the precipitate, and add the washings to the bulk of the filtered liquid; to which add excess of ammonia and carbonate of ammonia: by these reagents the small quantity of lime and the excess of baryta in solution are precipitated. The solution must now be filtered, evaporated to dryness, and ignited. The dry residue consists of chlorides of potassium and sodium, which must be weighed, then dissolved in water to which a little hydrochloric acid is added, then excess of chloride of platinum, and the whole evaporated to dryness in the water bath; alcohol is now added, and the whole thrown on a small filter. The yellow precipitate of platino-chloride of potassium on the filter is washed with alcohol until the latter passes off colourless. The filter and its contents are then dried and weighed. Every 100 parts of platino-chloride of potassium correspond to 30·56 parts of chloride of potassium. The quantity of chloride of potassium thus obtained is deducted from the weight of the mixed chlorides of sodium and potassium as obtained above; the difference will be the amount of chloride of sodium. Every 100 parts of chloride of sodium correspond to 53·28 of soda, and every 100 parts of chloride of potassium to 63·25 of potash.

*Determination of Sulphur.*—Dissolve 100 grains of the ore in either of the manners already described, separating the silica; in this case, however, a little nitric acid must be added to the hydrochloric acid previous to its mixture with the ore. To the filtered solution, made somewhat dilute, add excess of chloride of barium, and allow to stand in a warm place for a few hours. Collect the precipitate of sulphate of baryta on a filter, wash, dry, ignite, and weigh. Every 100 parts correspond to 13·79 parts of sulphur.

*Determination of Carbonic Acid.*—The most convenient apparatus for the determination of this gas is that invented by Fresenius and Will, of which the following is a description. Fig. 79 shows its construction. *A* is a large flask of about two ounces capacity, in which the decomposition of the carbonate is effected: *B* a somewhat smaller flask, containing strong sulphuric acid: both are supplied with doubly pierced corks, for the reception of the three tubes *a*, *c*, and *d*. The tube *a* is confined to the flask *A*, being immersed below the level of the fluid: in the same manner, *d* is only connected with the flask *B*, and only extends just below the cork. Lastly, the tube *c* enters the neck of *A* on the one side, but does not extend further, and, by a double bend, is brought into connection with *B*, which it enters, dipping into the sulphuric acid. The mouth of *a* is closed with wax during the experiment, so that no orifice is left in the whole apparatus but the mouth of the tube *d*.

FIG. 79.



The large assay balance, represented by fig. 11, is admirably suited for weighing this apparatus.

100 grains of the ore are introduced into the flask *A*, which is then filled with water to about one-third; the apparatus is closed by the wax stopper, and brought into equilibrium on the balance by a counterpoise. The decomposition of the carbonate under examination is now induced by sucking out a small quantity of air with the mouth from the tube *d*. The air is thus drawn not only from *B*, but also from *A*, both flasks being connected by the tube *c*; bubbles of air are therefore seen passing from *A* through the sulphuric acid; and in order to restore the equilibrium of pressure, a small quantity of sulphuric acid is forced from flask *B* into flask *A*, where coming in contact with the carbonate under examination, it decomposes it; and the carbonic acid evolved with effervescence in *A* can only escape by the tube *c* into the flask *B*, whence it must pass through the remainder of the sulphuric acid and the tube *d* into the air. This sulphuric acid condenses with great energy all the aqueous vapour, and retains everything that the current of gas might possibly carry with it. When the operation of removing a small quantity of air by the mouth, and the consequent addition of corresponding quantities of sulphuric acid to the contents of flask *A*, have been repeated until no more effervescence ensues, the decomposition is complete.

There is still, however, a portion of carbonic acid remaining in the apparatus which was previously filled with air, and some still clings to the solution in the flask *A*, which by this time has become cold. Both must be removed before the apparatus is re-weighed. For this purpose, by suction, as in the commencement, at *d*, so much sulphuric acid is caused to pass over at once as will give rise to a considerable elevation of temperature in *A*, by which means the carbonic acid in solution is evolved, and with it that portion still clinging to the other parts of the apparatus. By removing the wax stopper *b*, the mouth of *a* is opened, and air may then be drawn through the apparatus from *d* until all the carbonic acid is expelled. Here, too, all the moisture which is removed by the current of air from *A* will remain in the sulphuric acid in *B*. When the whole apparatus has cooled it is placed upon the scale, and the amount of carbonic acid is ascertained by the weights which must be added to re-establish the equilibrium.

*Determination of Water.*—Weigh 100 grains of the ore, and ignite for a quarter of an hour in a lightly covered platinum crucible. When cold, weigh the ignited ore; the loss is carbonic acid and water. Deduct the amount of carbonic acid previously obtained from the total loss, and the remainder represents the quantity of water.

## BLOWPIPE REACTIONS OF IRON ORES.

**IRON ORES.**—*Sulphuret of Iron (Magnetic Pyrites).*—*Alone*, undergoes no change before the blowpipe. In the *open tube*, gives sulphurous acid. *On charcoal*, becomes red in the outer flame, and is changed, by roasting, into an oxide of iron.

*Common Pyrites.*—*Alone*, in the matrass, exhales an odour of sulphuretted hydrogen, whilst sulphur is eliminated. *On charcoal* it behaves like magnetic pyrites.

*Mispickel, Arsenical Pyrites.*—*Alone*, gives first a red sublimate, which is sulphuret of arsenic, then a black; and lastly, in a strong fire, metallic arsenic sublimes.

*On charcoal*, mispickel gives a thick smoke of arsenic, then fuses, exhaling the odour of that metal. If the mispickel contain cobalt, it can be detected after well roasting the ore, and fusing the residue with borax or microcosmic salt; after cooling, the glass takes the characteristic colour of cobalt.

*Magnetic Oxide of Iron, and Oxide of Iron*, behave as already described.

*Carbonate of Oxide of Iron*, heated in the matrass, gives no water. Some species decrepitate strongly. Exposed to a gentle heat, it blackens, and gives oxide of iron, very attractable by the magnet.

*Chromate of Iron.—Alone*, undergoes no alteration. With borax and microcosmic salt, the solution is slow but complete. The characteristic colours are alone apparent when the bead is hot; but as soon as it cools, the fine green of chromium makes its appearance. This reaction is most intense when the substance is treated in the reducing flame, and appears in all its lustre by the addition of tin.

*Hydrated Oxide of Iron* gives water in the matrass, and leaves red oxide after fusion with microcosmic salt; it gives with tin some traces of copper.

**OXIDES OF IRON.**—*Alone*, undergo no change in the oxidising flame; but in the reducing flame the first two blacken and become magnetic.

*With borax* they give a dull red glass in the oxidising flame, which brightens on cooling, and finally takes a yellowish tint, or even becomes colourless on cooling. If the bead contain a very large proportion of oxide, it is opaque in the liquid state, and, on cooling, becomes a dull impure yellow. In the reducing flame, it becomes bottle-green, and, if the reduction be forced to the highest possible extent, assumes a lively bluish-green tint, exactly like protosulphate of iron. Tin very much accelerates the reduction of the higher oxides to the state of protoxide.

*With Microcosmic salt* they behave as with borax, but the green colour disappears more completely, and may be entirely got rid of by the application of tin.

*Soda* does not dissolve the oxides of iron, but causes them to be absorbed by the charcoal, in which they are easily reduced, and may be obtained as a grey, magnetic, metallic powder.

The following method for distinguishing protoxide of iron from the sesquioxide, is given by Chapman in the 'Chemical Gazette:'—

A very minute quantity of oxide of copper is to be dissolved in a bead of borax on the platinum wire until the glass acquires a slight coloration; the substance under examination now being added to it, the whole is subjected, for an instant only, to the reducing flame, when, if protoxide of iron was present in the assay matter, the oxide of copper will be reduced to suboxide, forming small red spots or streaks which become visible as the glass cools. The oxide of iron is converted into sesquioxide at the expense of the oxygen of the copper.

In the above experiment, if the glass were exposed for too long a time, the oxide of copper might be reduced, even if the substance under examination contained only sesquioxide of iron, as this would be converted by the flame into protoxide, and thus act, as before stated, on the oxide of copper; and if, furthermore, this latter substance were contained in too large a quantity in the borax glass, it might become reduced by the sole action of the flame, and thus give rise to an error. To obviate, therefore, all doubt as to the presence or absence of protoxide of iron, the same authority proposes that the operation should be conducted in a different manner, which gives certain results.

The borax bead must be coloured by a sufficient quantity of oxide of copper to render it of a fine blue colour, but transparent when cold. To this the substance under examination in powder must be added, and then exposed for a moment, or until the iron compound begins to dissolve in the oxidating flame.

If sesquioxide of iron alone be present, the glass will remain transparent, and of a green or bluish-green colour; but if, on the contrary, the iron is in the state of protoxide, the glass, on cooling, will be marked with opaque red patches, due to the reduction of oxide of copper into suboxide, as before explained. Care must be taken not to continue the blast too long, otherwise the suboxide of copper might again be oxidised, and the whole of the protoxide of iron be converted into sesquioxide. After one or two trials, however, no error can possibly arise. The reactions are not prevented by the presence of silicic or other acids.

# CHAPTER X.

## THE ASSAY OF COPPER.

IN the assay of copper by the dry way, all minerals and substances containing that metal may be divided into three classes.

### CLASS I. *Comprises Sulphuretted Ores or Products, with or without Selenium, Antimony, or Arsenic.*

| | | | | | |
|---|---|---|---|---|---|
| Copper glance, | $Cu_2S$, | containing | 79·7 | p. c. | of copper. |
| Chalcopyrite, | $Cu_2S,Fe_2S_3$, | „ | 34·4 | „ | „ |
| Erubescite, | $3Cu_2S,Fe_2S_3$, | „ | 55·7 | „ | „ |
| Bournonite, | $3Cu_2S,SbS_3+2(3PbS,SbS_3)$ | „ | 12·7 | „ | „ |
| Fahlerz, | $4(Cu_2S,FeS,ZnS,AgS,HgS).(SbS_3,AsS_3,Bi_2S_3)$ | „ | 30—48 | „ | „ |
| Covelline, | $CuS$, | „ | 66·7 | „ | „ |
| Wolfsbergite, | $Cu_2S,SbS_3$, | „ | 24·9 | „ | „ |
| Domeykite | $Cu_6As$, | „ | 71·6 | „ | „ |
| Copper regulus, Copper speiss, &c. | | | | | |

### CLASS II. *Oxidised Ores and Products.*

| | | | | | |
|---|---|---|---|---|---|
| Red copper, | $Cu_2O$, | containing | 88·7 | per cent. | of copper. |
| Malachite, | $2CuO,CO_2+HO$, | „ | 57·3 | „ | „ |
| Auzurite, | $2CuO,CO_2+CuO.HO$ | „ | 55·1 | „ | „ |
| Cyanosite, | $CuO,SO_3+5HO$, | „ | 25·3 | „ | „ |
| Phosphate of copper, | | „ | 30—56 | „ | „ |
| Arseniate of copper, | | „ | 25—50 | „ | „ |
| Chromate, Vanadate, and Silicate of Copper; Slags, &c. | | | | | |

### CLASS III. *Copper and its Alloys.*

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The different methods of assaying copper are more numerous than those for any other metal. They are, in some cases, very similar to each other, and in others based upon very different principles.

These methods may be classified in the following manner:—

### A. Assay in the Dry Way.

#### *a. For rich Ores and Products of Class I.*

1. English Copper Assay.
2. German Copper Assay.

#### *b. For poor Ores and Products of Class I.*

1. Fusion of the ore, &c., to a crude regulus, and further treatment of the same according to *a* 2.
2. Fusion of the roasted ore with reagents to collect the copper (lead, antimony, or arsenic), and refining the crude copper.

#### *c. For Ores and Products of Class II.*

1. *Reducing and solvent fusion*, with or without collecting agents (antimony, arsenic, lead), for the copper, after the ore has been roasted, if necessary (in the case of cyanosite and arseniate of copper) with coal dust, graphite, or carbonate of ammonia.
2. *Concentration smelting* with pyrites to a regulus, which is then roasted and smelted to crude copper with or without collecting reagents for the copper.

#### *d. Copper and its Alloys. (Class III.)*

Refining with lead on the cupel, or with borax on the refining dish, with or without the addition of lead, antimony, or arsenic.

### B. Assay in the Wet Way.

#### I. *For Substances rich in Copper.*

- *a. Kerl's modified Swedish Assay.*
- *b. Assay of copper by metallic zinc.*
- *c. Colorimetric Copper Assays.*
    1. Heine's method.
    2. Jacquelin's and von Hubert's method.
    3. Müller's assay with complementary colorimeter.
- *d. Volumetric Copper Assays.*
    1. Pelouze's method.
    2. Dr. Penny's method.
    3. Kunsel's method.
    4. Parkes' and Mohr's methods.
    5. Schwarz's method.
    6. Brown's method.
    7. Fluk's method.
    8. Fleitmann's method.

*e. Other Copper Assays.*
  1. By Levol.
  2. By Robert and Byer.
  3. By Rivot.
  4. By Wolcott Gibbs.

*f. Copper Assays by the blowpipe.*

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## A. ASSAY IN THE DRY WAY.

### *a. For rich Ores and Products of Class I.*

### I. ENGLISH COPPER ASSAY.

M. L. Moissenet has given in the 'Annales des Mines'[*] a very complete description of the English method of assaying copper by the dry way. The following is from a translation by Mr. W. W. Procter.

Each of the large Swansea copper works keeps an assayer at Cornwall, whose duty it is to determine the richness in copper of all the lots of minerals of the county sold every Thursday at the *Ticketing*, and of all the samples of foreign minerals and copper products which may be useful to the smelter.

It may be asserted that in the course of a year there are but a few copper mines being worked on the surface of the globe of which some sample has not been addressed to the master assayers of Cornwall, and in the same interval each laboratory has made not less than 8,000 to 10,000 assays.

In consequence of the great number and variety of the matters to be treated, and the necessity of having a prompt answer, we see the necessity of a simple and expeditious method. It is natural, then, that the dry way should be preferred to the wet way; besides, other considerations support this choice.

The copper being obtained in the state of prill or metallic button, the impurities (generally tin, antimony, &c.) are thus made evident, and the hammer soon proves the quality

[*] Vol. xiii. p. 183.

of the metal which we ought to expect to obtain by metallurgic treatment. As for the accuracy of the method, as far as regards the whole of the metal obtained, I shall revert to this later on. I would, however, observe that, within certain limits, the method would not be less practical on account of being inexact; for we must not forget that it has chiefly for its object to teach the smelter the *value* of the mineral even more than its true richness.

For example, if we get too low an assay from a sample of 2 or 3 per cent., we should only from this assent to the opinion of the metallurgist, whose interest it is not to work upon very poor minerals. The same remark will apply to the case of minerals very antimonial, &c. Besides, in the description of the method we shall discover the principal phases of the Welsh process; so that it is more just to consider the Cornish assay as a metallurgy on a small scale than as a scientific laboratory method. From thence result also the necessity of long practice and the almost uselessness of theoretical knowledge for those who purpose employing this method alone.

Sir Henry De la Beche ('Report on the Geology of Cornwall,' &c. p. 595), in giving a sketch of the method, declares it to be rather rough and uncertain, and fails not to add at the conclusion a translation of a passage relative to the assay of copper pyrites from M. Berthier's treatise on assays by the dry way.

These drawbacks upon the scientific value of the English method cannot injure the power of facts; they constitute but another reason which we may have for giving an account of the manner in which the first basis of the valuation of the greater part of the copper minerals has been fixed since so long a period.

*Division Adopted.*—The rather complex operations through which we have to pass will be better apprehended, I think, by explaining in succession—

1. The order of the operations, the nature and influence of the fluxes employed, the kind of products obtained (reactions).

2. The manipulations to which each operation gives rise,

the furnaces and apparatus used, the characters of the principal products during the chief phases and at the end of each (manipulations).

I shall add to these—

3. Some information upon the influence of the principal foreign metals (tin, antimony, zinc, lead), and upon the treatment of some special coppery matters.
4. Summary considerations on the result of the English method compared with those of the analysis by the wet way.

### SECT. I.—REACTIONS.

At the very outset we distinguish two kinds of assays.

1. The roasted sample.
2. The raw sample.

The first only applies to cupreous pyrites or to samples essentially formed of it—that is to say, which contain sulphur in excess: the process begins by a roasting.

In the raw assay we dispense with the roasting; we have recourse to the addition of reagents, either oxidising or sulphurising, according to the minerals; we endeavour to place them by these mixtures in the condition of a properly roasted pyritic mineral.

From this point, at least in general, the operations become identical. They consist in—

1. Fusion for regulus (regulus).
2. Calcining the regulus (calcining).
3. Fusion for coarse copper (coarse copper).
4. One or two fusions with fluxes (washings).
5. Trial by striking with a hammer, last refining (testing, refining).
6. Treatment of slags for prill.

All the slags except those of the fusion for regulus have been preserved. The fusion No. 6 gives a small supplementary button of copper, which again undergoes, if necessary, one or two washings.

As I have said, the roasting is used only for pyrites. I shall return later on to the duration and the circumstances of this operation. Its evident aim is to drive off the excess of sulphur, so as to cause the whole of the copper, with a part only of the iron which abounds in the pyrites, to pass into the state of sulphide at the time of the fusion for regulus.

### I. *Regulus.*

1. *Pyrites.*—The fusion for regulus of a properly roasted pyrites is made by mixing with it equal volumes of the three fluxes, borax, fluor-spar in powder, lime slaked in powder, of each one ladle, and covering the mixture with a layer of moist common salt. The matters composing the gangue of the roasted mineral consist principally of quartz, silica, and in general of more alumina and magnesia than lime; oxide of iron, resulting from the roasting of the pyrites is also present.

The borax only serves to give fusibility, the fluor-spar contributes to the same end by forming a fluosilicate. I do not think that it otherwise plays an important part in the decomposition—that is to say, that there may be a production of fluoride of silicon and calcium; for this last base is added here in considerable proportion, so as to form immediately a silicate which may combine with the fluoride of calcium.

The peroxide of iron being reduced to pass into the slag, and the different metallic oxides to pass into the regulus, yield oxygen, which reacts on the remaining sulphur. The disengagement of sulphurous acid which results from this, joined to the water contained in the fluxes, justifies, to a certain extent, the use of a bed of common salt, designed to prevent the boiling over. Besides this, the common salt being without action on the metallic sulphides, does not here produce those important effects which I shall point out in the later fusions.

If the pyrites appear insufficiently roasted, we must add a little nitre, the oxidating action of which again gives off sulphur; the opposite case, that of a roasting too much prolonged, is rare; we remedy it by the addition of sulphur and tartar, as I shall indicate for other sorts of minerals.

2. *Very poor Pyrites.*—In a very poor pyrites—that of

Bear Haven, in Ireland, for example—the proportion of sulphur does not require us to have recourse to the roasting; we employ the three fluxes and one ladle of nitre.

3. *Variegated Copper Ore.*—Peacock ore contains less sulphur in proportion to the copper than pyrites; we also fuse with a little nitre.

4. *Sulphide of Copper.*—The sulphur is here insufficient. We add together sulphur ½ to 1 ladle, according to the valuation; tartar ¼ to ½ ladle—that is to say, half the volume of the sulphur. The tartar is a powerful reducing agent, and is supposed in small quantities to favour the action of the sulphur by preventing its disengagement as sulphurous acid by the oxidating matters in the mineral; but if used in excess, it acts as a desulphuriser, as well by its carbon as by its alkali.

5. *Carbonated Minerals.*—The addition of sulphur and carbon is evidently still more necessary here.

6. *Native Mixture*: ⅔ sulphide copper, ⅓ pyrites.—We add, in this case, nitre for the pyrites, and sulphur and tartar for the sulphide of copper; although these reagents appear sure to neutralise each other, it is possible that their simultaneous employment may be logical. The nitre probably decomposes the pyrites, which would, without it, fuse and give a very ferrous regulus, whilst the free sulphur would be of little use, on account of the sulphide of copper. Be this as it may, this is the plan adopted.

During the progress of the fusion for regulus we have still to introduce other matters, some accidentally, and others in all cases.

If a blue flame persists in escaping from the crucible, an index of the formation of sulphurous acid, we project into it sulphur 1 ladle, tartar ½ a ladle. When the fusion appears almost finished, in order to render the bath more liquid, and to facilitate the collection of the button, we throw in a little dried salt and a flux composed beforehand of lime, a little fluor-spar, and a very little borax—that is to say, of the elements in different proportions of the mixture introduced originally.

The regulus obtained is composed principally of copper, iron, and sulphur. I shall return to the aspect and the richness which it ought to have according to the minerals treated.

### II. *Calcining.*

The calcination of the regulus is one of the most important operations; it ought to be quite complete.

### III. *Coarse Copper.*

To the calcined regulus is added—nitre $\frac{1}{4}$ ladle, borax $\frac{1}{2}$ ladle, charcoal $\frac{1}{8}$ ladle, dry salt 1 ladle. These quantities remain the same, whatever mineral may be assayed—tartar 2 ladles. Case of medium richness. Covering of moist salt, 2 ladles.

The nitre is designed to burn the sulphur which may have escaped the calcining, and to ensure the passage of the easily oxidisable metals, especially of iron, into the slag in the state of oxides. It is besides in too small proportion to act upon the copper, especially in presence of reducers whose effect is certainly later than the deflagration of the nitre.

The borax plays simply the part of a flux.

The dry salt has for its object to give fluidity to the slag. Unfortunately, if the addition of the salt attains this object, it also determines from this operation a sensible loss of copper by carrying it away with the saline vapours. I shall insist upon this point in describing the washing.

The charcoal and the tartar are especially the important reagents in the fusion. The tartar, at the same time that it is one of the most energetic reducers, is also a flux and a desulphuriser. Its use is, then, perfectly justified here, only the proportion of tartar added ought to be regulated according to the quantity of copper, which the *weight* and *aspect* of the regulus permit the experienced assayer to estimate sufficiently close; an excess of tartar would reduce the foreign metals, and produce in consequence a very impure coarse copper.

When the fusion appears complete, we throw in a pinch of white flux,* which gives fluidity to the slag, and determines by its partial decomposition, from which a disengagement of carbonic oxide results, a stirring up of the materials. These two effects facilitate the collection of the metallic button. The carbonate of potash begins also without doubt from this operation to refine the metal a little by attacking the iron, zinc, and tin already reduced. M. Berthier ('Essai par la Voie Sèche,' vol. i. p. 393) points out this reaction:—'A part of the carbonic acid which it contains being decomposed and changed into carbonic oxide, a compound is formed consisting of alkali, carbonic acid, and metallic oxide, &c.'

Lead, copper, and antimony are not attacked.

### IV. *Washings.*

In the operation of washing we put into the crucible at the same time as the coarse copper the following fluxes:—White flux, 1 ladle; dry salt, 2 ladles.

It is evident that the white flux is here employed as an oxidiser of the foreign metals, and with a view of the application of the above-mentioned reaction.

As for the salt, it is both useful and injurious. If it were only used with the view of augmenting the fluid mass so as to preserve the metal from contact of air, &c., it would be advantageously replaced by an excess of white flux; but it can form with the arsenic and antimony which the copper has retained in the form of arseniuret and antimoniuret, volatile chlorides. Common salt is, then, to be regarded as one of the principal agents of purification put in operation by the English method. On the other hand, the loss of copper which arises from the carrying off of this metal by the vapours of common salt cannot be doubted. M. Berthier has found that by heating equal weights of copper and salt until the complete volatilisation of this last, 3 per cent. of the metal is carried off.

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* This white flux is prepared in the laboratory by mixing in a mortar, tartar 3 volumes, nitre 2 volumes, salt a little, then determining the combustion by the introduction of a red-hot iron rod, which is turned round until the matter ceases to deflagrate.

In the event of the coarse copper appearing too impure, we take care to add a little nitre. According to the appearance of the button we recommend the washing or not.

### V. *Testing, Refining.*

The button of metal is flattened on an anvil. We thus recognise tin by the hardness and antimony by the brittleness of the alloy. The button is then put alone in the crucible. When it presents a proper appearance—that is, when the edges assume a bright colour, the centre, which the assayer calls the eye, being dark—we hasten to put into the crucible the fluxes, which are the same as for washing, only taken in rather smaller quantity.

In general, when we have operated well the button obtained is of a fine colour, and is regarded as pure; if we have passed the eye, it is covered with a layer of red oxide; if, on the contrary, we have put in the fluxes too soon, the button is dull.

It is easy to give an account of the reactions which take place during the refining, and which differ a little from those of the washing.

In heating the button alone in the air in the crucible, it is intended to submit it to an oxidation, which ought to act sufficiently on all the foreign metals more oxidisable than copper without acting too much on this last. The proper point is indicated by the appearance of the eye: the projection of the fluxes puts an end to the atmospheric oxidation, and determines the scorification of the oxides which expel part of the carbonic acid of the carbonate of potash, for which they substitute themselves, and give rise to triple compounds of metallic oxides, alkali, and carbonic acid.

The oxides of lead, tin, iron, and zinc comport themselves thus. When we have passed the eye, there has been a considerable formation of oxide, which leaves the button reddened, as I have indicated. At the same time the slag is strongly coloured red or green. If, on the contrary, the fluxes have been thrown in in too great haste, the oxidation has been insufficient, and then the refining just falls back upon the preceding operation of washing—an operation less efficacious and even without result in the case of lead and antimony.

As for the physical phenomenon of the eye, perhaps it corresponds to the very short instant when the oxides, less dense than the copper, are concentrated at the top of the button, and there make a dark spot before attaining a temperature sufficiently elevated to acquire the brightness of the metal itself.

I shall add that the minerals of Cornwall, generally more impure than foreign minerals, require a notably longer time for the appearance of the eye.

*Extra Accidental Washing.*—More often the refining gives a definite product, put aside to be weighed with the prill extracted from the slag; let the button be clear, burnt, or dull. Even if the metal appeared too impure we would not recommence the refining, but would have recourse to an extra washing by putting at once into the usual crucible besides the button and the usual fluxes, the slag from the refining.

### VI. *Slags for Prill.*

All the slag from the fusion for coarse copper inclusively having been preserved, we fuse them altogether with—

| | | |
|---|---|---|
| Tartar . . . | 1 ladle | Simple reducing mixture. |
| Charcoal . . . | traces | |

We obtain a small globule variable with the circumstances of the different operations which have allowed more or less copper to pass into the slag. If the prill is not very small, and its appearance indicates a metal not sufficiently pure, we submit it to one or two washings, as above.

## SECT. II.—MANIPULATIONS.

The sample, which has been taken with the utmost care, arrives at the laboratory rather coarsely powdered, still wet, and wrapped in strong packing paper: the paper is opened and placed near a furnace on the cast-iron plate which covers it; the drying is rapidly done there.

The first question is to discover the kind or kinds of minerals, so as to employ the warm or raw sample.

For this purpose we throw one or two large pinches of the mineral into a flat-bottomed copper dish, and we wash it very easily by putting in water several times and giving a rotatory motion to the matters, at the same time that we incline the dish so as to cause the muddy parts to run from the gangue. The small metallic fragment remains distinctly visible, and we can often discern by simple inspection the presence of foreign metals.

We weigh 400 grains of the dried mineral, a quantity upon which the assay is made.

The crucibles used in Cornwall are of three sizes:—

1. Large.
2. Large second.
3. Small second.

The small seconds have externally the internal dimensions of the large, into which they fit as into a nest; the first and third are sold the one in the other, and called nested. They are the most used.

The large serve for the roasting and the fusion for regulus, the small second for calcining the regulus and all the fusions which follow.

The large seconds are only employed in place of the former when we have to treat a very large regulus.

The crucibles are of a kind rather wrinkled, and as if fused superficially, they present the appearance of coarse stoneware pottery. Their form, moderately wide, permits us to make use of them successively for the roasting and the fusion for regulus, and gives them sufficiently great stability in the fire of a wind furnace. They are besides very resisting. They are made at Truro and Redruth.*

The wind furnace has for its principal dimensions—

| | | Inches |
|---|---|---|
| Length from front to flue | | 10 |
| Breadth | | 8 |
| Depth to the bars | | 14 |
| Opening of the flue | length | 8 |
| | height | 2 |

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* Mr. Juleff, of Redruth, is considered to make the best crucibles. Each laboratory uses 1,500 dozens annually. A lid is never used for the crucible.

A sufficiently large space is reserved underneath the fire, where the ashes accumulate without inconvenience, but opening only by a framework contracted so as not to allow too free an access of cold air.

The furnace serves either for roastings or for fusions; in the latter case we cover it with two mounted bricks, very easy to manage, and allowing to only half open it when we wish to inspect the contents of the crucibles. We can conduct ten roastings at once; the crucibles are marked by a brush with colcothar mixed with water. The furnace having been recharged with coke, we put the crucibles on the top, and after a few minutes, the substances beginning to get warm, we stir them by means of iron rods. Each crucible receives a rod which we leave standing there (leaning against the chimney) during the whole period of the roasting, so as to avoid the loss which would take place if we withdrew the rod. From time to time we renew the surfaces by lightly taking hold of the rod with the left hand by the upper end, whilst the right forefinger and thumb make it turn at once upon itself and round the crucible.

The duration of the warming varies essentially with the nature and the richness of the mineral; it is never less than six or seven minutes, and may reach half an hour. When from the *sandy appearance* of the matters we consider the operation finished, we withdraw the crucible, raise the iron rod with care, and expose the crucible to the air, allowing its contents to cool slowly. The roasting has succeeded when the surface has the brown red colour of oxide of iron and the bottom only is black. In this case we proceed to the fusion for regulus by simply adding the three fluxes (borax, fluor-spar, and lime); if the bottom of the crucible appeared too black, we ought to complete the oxidising action by the addition of a little nitre.

### *Fusion for Regulus.*

The different substances above indicated are taken from the box with a slightly concave ladle of 1⅜ diameter, then mixed in the crucible with a stirring-knife. We ought to allow the heat of the wind furnace to fall and to recharge, so as to have a gentle fire at the commencement of the fusion for regulus. The crucibles are placed upon the coke, and supported against the walls of the furnace, which we then close with the two bricks. After about a quarter of an hour, we open the front brick so as to observe the progress of the operation; it is at this stage that we throw in the sulphur and tartar into those crucibles from which a blue flame is disengaged. Some minutes later—that is to say, nearly seventeen minutes from the commencement, we add the salt and the flux destined to collect the regulus; then (twenty minutes from the beginning) we run into a metal mould, not greased.

We make, in general, several fusions at once—four, for example; we have in consequence two moulds into which we pour the contents of the crucibles in an adopted order, so as to avoid all confusion. The matters, very rapidly solidified, are detached simply by a blow, and fall in order on a metal plate fixed in front of the laboratory window. We immediately seize them with the copper tongs, put them into a basin of the same metal, and immerse them for a moment in cold water, where it is important not to leave them too long. This immersion allows us then to separate very easily the slag from the button of regulus, itself very brittle. For this purpose the fluxions are put on the metal plate, and by means of a hammer we strike with care all round the slag, which breaks off pretty cleanly. We hasten to detach from the surface of the regulus the slag which may remain adherent, using a small hand chisel, without the hammer. The slags are broken, and if we find any prills of regulus they are added to the principal button. Sometimes in these breakings, and especially in those analogous for the last fluxings, we surround the substances by an iron ring, placed on the metal plate, so as to avoid loss of splinters. In a general way, the slags of the fusion for regulus are rejected. We shall see further on how it may become necessary to flux them again when the mineral contains blende.

The aspect of the regulus is characteristic, and it is easy to arrive at a pretty close estimation of its richness, and consequently of the degree of success of the operation, by simple inspection of the regulus.

No. 1. A regulus very poor (coarse), that is to say, too much charged with iron, is bronzed and dull; the operation following would not be able to carry off the excess of iron. at least without a corresponding loss of copper. A like regulus evidently results from an imperfect warming, or from an excess of sulphur, or from an insufficiency of nitre, as the case may be.

It contains less than 40 per cent. of copper. There is nothing for it but to reject it.

No. 2. A regulus of good appearance is in general bronzed but rather shining; it appears finer. Its richness varies from 40 to 60 per cent.

No. 3. From oxides, carbonates, and from some minerals charged with impurities (SnSb) we desire to obtain a fine bluish button of a greater richness—65 to 75 per cent. We perceive, indeed, that for oxides and carbonates, to which we have only to add sulphur, and which also by their nature do not, like pyrites, contain combined iron, it is easy to obtain a richer regulus without fearing any loss of copper. As for the stanniferous and antimonial minerals, I shall return to them further on.

No. 4. In every case a regulus, the richness of which rises to 80 per cent., and of a very shining grey blue appearance, ought to be rejected, its richness indicating the loss of a certain quantity of copper left in the slag.

Here is, in the preceding order, the result of the analyses of four buttons whose description agrees with that which I have just given, excepting, perhaps, No. 2, whose fracture is rather reddish:—

| No. | | Copper | Iron | Balance; sulphur & traces of foreign metals |
|---|---|---|---|---|
| 1. | Coarse, to be rejected . . . | 36·00 | 32·90 | 31·10 |
| 2. | Good in general (rather too fine) . | 60·00 | 14·70 | 25·30 |
| 3. | Good for a carbonate, &c. . . | 65·60 | 10·50 | 13·90 |
| 4. | Too fine, to be rejected . . . | 80·16 | 2·10 | 17·74 |

If we compare these products with those obtained in the metallurgy of copper by the Welsh method, we find (Le Play, 'Annales des Mines'):—

| Matts of the operations.<br>II. V. IV. VIII. | | Copper | Iron | Different metals | Sulphur | Total of Diff. M. & S. |
|---|---|---|---|---|---|---|
| II. | Coarse matt (fusion of poor minerals, raw or calcined)<br>$3Cu_2S+Fe_2S_3+4(Fe.\ dif.\ met)S$ | 34·6 | 34·1 | 1·5 | 29·8 | 31·3 |
| V. | Blue matt (fusion of the calcined coarse matt with minerals of mean richness)<br>$0{\cdot}8Cu+3Cu_2S+2(Fe.\ d.m.)S$ | 57·2 | 18·5 | 1·0 | 23·3 | 24·3 |
| | Reddish variety, *matte mince*<br>$1{\cdot}3Cu+3Cu_2S+2(Fe.\ d.m.)S$ | 61·6 | 15·8 | 0·6 | 22·0 | 22·6 |
| IV. | White matt (fusion of the calcined coarse metal with rich minerals, carbonates, and oxides) | | | | | |
| | Metal—very pure type | 77·4 | 0·7 | 0·9 | 21·0 | 21·9 |
| | very blue variety | 64·8 | 9·0 | 3·6 | 22·6 | 26·2 |
| | mean | 73·2 | 6·3 | — | 20·5 | — |
| | $8Cu_2S+FeS$ | | | | | |
| VIII. | Matt (roasting of extra white matt VII.)<br>$0{\cdot}2Cu+Cu_2S$ | 81·1 | 0·2 | — | 18·5 | — |

These numbers show the evident analogy, the identity almost, of the products of the laboratory and those of the works; we may sum up by saying that the regulus ought to be richer than coarse metal, and in the case of ordinary minerals to approach if not to attain (as in the case of sample No. 2) to the composition of blue metal.

For carbonated and oxidated minerals we arrive directly at the very bluish variety of white metal.

Finally, in no case must we arrive at a button as rich as regulus matt.

### *Calcining the Matt.*

The matt is pounded fine in a bronze mortar; we avoid loss of fragments by means of a perforated cover and a cloth which surrounds the pestle. To facilitate the pulverisation, and avoid the sulphuret greasing, we add in the mortar a small piece of coke. The pounded matt is carefully turned upon a sheet of paper, the mortar wiped out with a hare's foot, and the substance put into a small second or large second crucible. The calcining is conducted as the warming of a mineral; it generally lasts longer, for the expulsion of the sulphur is to be as complete as possible. It demands the most minute care to regulate the fire so as to avoid all agglomeration, and to stir almost continually. When the matter adheres to the rod, we withdraw the crucible for a moment; this inconvenience is chiefly produced, if we have not detached the slags sufficiently from the matt; the calcining is then much longer, the flames remain blue a long time, and the fumes which are disengaged have an odour which is not purely that of sulphurous acid. When the fumes and the odour cease, and the matter has taken a sandy appearance, we raise the heat; then withdraw it, and allow to cool slowly in the air as for warming.

The mean duration of calcining is half an hour.

### *Coarse Copper.*

The fluxes above indicated are taken from the box No. 1, except the dry salt, which forms part of a second box called the refining flux. The ladle for this box No. 2 is a little larger than for the first; it has a diameter of ·044 m. At the beginning of the operation the furnace is well filled and lighted; the same fire ought to suffice for all the following fusions, which it is very important to conduct with great rapidity. After a moment, and if there is any frothing, we throw in some dry salt, which calms the ebullition. At the end of ten minutes, the fusion appearing complete, we throw in a pinch of white flux. A little after we withdraw successively each of the crucibles, pouring them carefully and by a single turn into each of the principal cavities of the metal mould. These moulds ought, this time only, to be greased with a cloth impregnated with suet. The crucibles are immediately put back again into the fire.

We detach the fluxion as previously, seize each one successively with the copper tongs, and plunge it for an instant into a basin full of water. The rest is effected as for the regulus, only the slags are preserved on the metal plate, and in the order in which we have detached them. The button of copper obtained appears more or less black; I have already indicated the influence of the tartar in excess.

### *Washings.*

We place the button and the fluxes in a large copper shovel, lengthened and narrowed at the end, called a scoop, and we pour them into the crucible, which is already at a red heat. As the fusion is made in five or six minutes, it would be inconvenient to prolong it on account of the loss occasioned by the carrying off of copper with the vapours of common salt. The tapping is made with care by pouring first into one of the large cavities, then as soon as the metal has fallen there we finish by pouring in the slag into one of the small lateral cavities. This last slag, probably rich in copper, is less fluid, and would adhere to the button, which would be difficult to cleanse. The two buttons being detached from the mould, we immerse the small one first, then finish as in the preceding operation.

### *Testing and Refining.*

The crucible has again been put back into the furnace—after the tapping; the button tried by the hammer is put into the crucible by means of the tongs. At the end of about three or four minutes it attains the colour of the vessel, the eye manifests itself, and we rapidly throw in the fluxes put into the scoop beforehand.

The tapping is made as for the washing, with the small button of slag kept apart.

In general we arrive at a button regarded as pure, clean copper; if not, as I have said, we proceed to an extra washing by adding exceptionally in the scoop the last slag obtained.

### *Prill.*

The crucible this time has been left out of the furnace; put into it all the slags, collected for this purpose from the metal plate into the scoop, and upon which we have put reducing reagents. The fusion lasts a quarter of an hour; pour all at once into the large cavity; before the cooling, by means of a transverse blow, get rid of the upper beds which are still liquid, and composed principally of common salt. Then operate as above. Collect the prill, which again undergoes, if necessary, a washing.

### SECT. III.—SOME MINERALS AND SUBSTANCES OF A SPECIAL NATURE—INFLUENCE OF FOREIGN METALS.

**STANNIFEROUS MINERALS.**—Most often we only perceive the presence of tin in a copper mineral when testing with the hammer, which reveals the nature of the bronze; when we proceed to the refining of such a stanniferous button it is impossible to obtain the characteristic eye; that is to say, the surface of the metal becomes quite clear, and we scarcely open the furnace when it again becomes obscure. We free it from tin by two or three extra washings. If we suspect tin from the known produce of the mineral, or the inspection of the sample in the basin, we endeavour to obtain a fine regulus, which is accomplished in the case of a warm sample by prolonging the calcining, and for the raw sample by putting in more nitre or less sulphur. It is clear that tin can only enter the regulus by virtue of the excess of sulphur necessary to the formation of the coppery matt, and that by restraining this excess of sulphur we diminish the chance of tin entering the button. The fine regulus ought to contain 70 to 75 per cent. of copper, as for the carbonated copper minerals.

**ANTIMONIAL MINERALS.**—Antimony is also detected in the testing; the metal being rendered very brittle. We then add one or two grammes (15 to 30 grains) of lead in the refining operation. There forms an alloy of lead and antimony heavier than copper, which is poured into the small cavity of the mould. When we suspect antimony, we act as for tin—that is to say, we produce a fine regulus, a most careful roasting expelling the antimony; then we have to make two washings, and in the second to add the metallic lead.

We cause, then, three influences to act with a view of expelling the antimony:—

1. Slow oxidation at a low temperature, disengaging antimony.

2. Repeated chloridations, from whence a formation of volatile chlorides.

3. Affinity of the lead and mechanical separation of the alloy.

**Zinciferous Minerals.**—One of the metals which is most troublesome is zinc. We recognise it by the appearance of the regulus and by its colour, which is that of blende. Once out of ten the regulus collects sufficiently to be able to detach it; in this case we pound it, add to it the slags, and borax 1 ladle, nitre ½ ladle. We fuse anew, and obtain a good regulus, for the nitre has caused the zinc to pass into the slag in the state of oxide.

Most often the zinciferous regulus does not collect, and there is nothing for it but to begin anew by making a very prolonged warming at least half an hour—for example, mineral of South Crenver—this is evidently what we should have done at first had we been aware of the presence of the zinc.

**Plumbiferous Minerals.**—Lead is not injurious, for it does not alloy with copper. The warming is also prolonged. Lead passes into the regulus, which facilitates the collection of the matter. In the last operation the lead easily passes into the slag; it also in case of need carries off antimony. Thus the copper obtained from lead minerals is most malleable.

### *Special Cupriferous Products.*

**Regulus of Chili.**—These are treated as those which we obtain by the fusion for regulus. Their richness, which rises to nearly 60 per cent., requires us to add much tartar in the fusion for coarse copper.

**Slags of Copper.**—To obtain regulus we add to the slag sulphur, tartar, and nitre; this last maintaining metals other than copper in the state of oxide in the slag.

**Old Copper.**—For turnings, waste of workshops, &c., yielding 97 to 98 per cent. by the assay, and containing, in fact, not much foreign matter except a little mixed dust or dirt, we take care first to glaze the crucible by fusing in it a little borax and nitre; then we treat the matters by a simple washing, the slags of which we work for prill. This last is often very considerable.

### SECT. IV.—SUMMARY CONSIDERATIONS—COMPARISON OF THE RESULTS WITH THE ANALYSIS BY THE WET WAY.

After this detailed account of the numerous operations which the metal undergoes before attaining the state of button and prill, it would, I think, be superfluous to insist upon the practical difficulty of the English method.

Nevertheless, in experienced hands, and in the case of daily practice, it is still a rapid method, allowing us to treat almost uniformly the different varieties of copper mineral, and at the least to remedy during the operation itself the impurities which show themselves.

As to the metallurgic accuracy, here is a small table showing comparatively the produce by the dry way (determined by a Cornish assayer) and that which I have obtained by the most precise methods of the wet way. It comprehends six samples, whose richness varies within sufficiently great limits.

| Nature of the sample and produce | Dry way D. | Wet way W. | Difference W - D. |
|---|---|---|---|
| Regulus of Chili . . . . . . | 56¼ = 56·250 | 58·40 | 2·150 |
| Green carbonate copper of Castile . | 9¾ = 9·750 | 11·52 | 1·770 |
| Variegated copper, Huel Damsel . . | 10½ = 10·500 | 11·30 | 0·800 |
| Pyrites, West Wheal Seton . . . | 8⅜ = 8·375 | 8·40 | 0·025 |
| „ United Mines . . . . | 8 = 8·000 | 10·38 | 2·380 |
| „ Devon Great Consols . . | 4⅝ = 4·625 | 5·60 | 0·975 |
|  |  |  | 8·100 |
| Mean difference . . | $\frac{\Sigma\,(W-D}{6}$ | . . | 1·350% |

By adding the result given by the last five minerals we find

$$\Sigma D=41{\cdot}25,\ \Sigma(W-D)=5{\cdot}95,\ \Sigma W=47{\cdot}20,\text{ and}$$

$$\frac{\Sigma W}{5}=9{\cdot}44.$$

By taking the ratio

$$\frac{\Sigma(W-D)}{\Sigma D} = 14{\cdot}42,$$

we see that we must add to the richness indicated by the Cornish assay about $\frac{1}{7}$th of that result, and by taking the ratio

$$\frac{\Sigma(W-D)}{\Sigma W} = 12{\cdot}60,$$

that the loss is $\frac{1}{8}$th of the copper if we consider a mineral of 9 or 10 per cent.

Without wishing to draw a conclusion altogether general from so small a number of analyses, I nevertheless think they suffice to show that the English method occasions losses always sensible and sometimes considerable. I think I may assert that upon the whole of the Cornish minerals whose mean richness varies from 6 to 7 per cent., the loss by the assay is not less than 20 per cent. of the contained copper, and that for certain pyrites of 3 to 4 per cent. it attains 30 and 40 per cent. of the metal.

The principal causes of these losses are,—(1st) The quantity more or less great of copper left in the slag of the regulus; (2nd) and especially the carrying away of copper by the vapours of common salt in the fusion for coarse copper, the washing or washings, the refining and the treatment of the slag for prill.

In consequence, I think they ought to bear principally on the oxidated minerals for which we make a rich regulus, and still more on the impure minerals, which besides a rich regulus, have undergone several washings. Thus the minerals of Algeria, grey copper, assayed some years ago at the School of Mines, have given a produce *much* higher than that indicated by the Cornish assayers.

### *Conclusions.*

In conclusion, the English method, as applied by the buyers and in their laboratories, certainly answers all their wants; but indicating results always lower than the real result, it would appear to be exercised to the detriment of the sellers.

The counter assays which are frequently made on account of mining companies and miners *tributors* can only be a proof of the good faith and of the truth of the figures announced by the buyer.

But we must not lose sight of the fact that the industrial methods of assay have for their object only to fix a basis of buying, and for this purpose it is not necessary that they should give a rigorously exact result; it is even logical that the loss in the assay should be proportioned to the loss in the treatment according to the greater or less impurity of the material.

Thus even if the Cornish companies should come to state in their products a richness of 1 or 2 per cent. greater, there would not result from this in reality any increase of *value* for their minerals, or, if we like, any advance of the buying price. This price is from other reasons far superior to those of copper minerals in foreign markets, and especially to that which is paid for American minerals at the works at Boston.

## II. GERMAN COPPER ASSAY.

This assay comprises the following operations:—

### 1. *The Roasting in the Muffle Furnace.*

From the correctly chosen, properly dried, and prepared assay substance, one centner is weighed out in duplicate for the assay. The weighed substance is so spread out on a roasting dish, that has been previously rubbed with chalk, rouge, or powdered manganese, that most of it lies towards the margin of the dish, and only a thin layer is found in the middle. The assay, which has also been previously well mixed with at least two parts by volume of charcoal powder, of twenty-five to forty pounds of graphite, is then placed in the dull red-hot muffle, and cautiously roasted till no more sulphurous or arsenious acid escapes.

The heat can be raised rapidly or only slowly, and the roasting finished in a shorter or longer time, according to the composition of the assay sample.

The presence of *lead*, *arsenic*, and especially *antimony*, makes special caution necessary, on account of the easy fusibility of their compounds. Such ores (*e.g. fahlerz*, *bournonite*, &c.), are roasted very gently at first, without the addition of coal, and afterwards powdered coal is used instead of graphite, as the ore thus roasts at a lower temperature. If, at the same time, as in many fahlerzen, *sulphide of mercury* is contained in the ore, the latter cakes together at a very low temperature, and at a greater heat evolves mercury with such rapidity that mechanical loss occurs. Such ores must be placed in the muffle while it is yet only moderately warm, in order to volatilise the mercury gradually, and be further roasted with coal only after the removal of the mercury. Coke and graphite work almost exclusively and continuously chemically, while charcoal dust has, at the same time, a disintegrating mechanical effect, since generally a large part of the latter is already burned before the ore has reached a temperature at which it can work upon it.

When the roasting ore has ceased to fume, and no longer gives forth any smell of sulphurous acid, it is ground in a brass or cast-iron dish, mixed with about twenty-five pounds of coal dust, and again roasted at a higher temperature till the odorous gases produced have disappeared. The assay is also sometimes calcined with tallow. If the ore is a very difficult one to roast, then, instead of the coal dust, a final addition of forty to sixty pounds of carbonate of ammonia is given, by which the sulphates (even the sulphate of lead) are decomposed with the formation of volatile sulphate of ammonia. Generally, a second grinding suffices. To secure a complete oxidation, it is necessary that the coal should be entirely consumed.

Arsenic can be removed, for the most part, by this roasting; still some basic arseniates are always formed. Antimony is more difficult to remove, and can only be in some measure driven off by careful roasting and frequent rubbing up. It remains behind as antimoniates. A residue of both these substances is less injurious than one of sulphur. The latter occasions in the reduction smelting a formation of sulphide of copper, the copper contents of which escape determination.

When no more fumes from the hot assay can be perceived by smell or sight, and the assay powder shows a constant, uniform, dull colour, and no more grains with metallic lustre can be seen while grinding it up, and finally when no more particles of coal or graphite can be detected, the roasting is finished.

Only with a completely roasted assay can it be counted upon that all, or as nearly all as possible, of the copper has been converted into oxide, and this must be accomplished if the result of the assay is to be correct. The copper which remains in the state of sulphide or sulphate is almost wholly lost, as by the later addition of alkaline flux it cannot be at all, or only very incompletely, reduced to metallic copper.

If the assay sample contains sulphates which are not converted into oxides by coal, graphite, and carbonate of ammonia (*e.g.* gypsum, baryta, &c.), it must be first smelted to a matt, which is then treated like a raw ore to be roasted. For this purpose one assay centner of ore, one centner of borax-glass, one centner of potash or soda glass, and ten pounds of colophony are well mixed together, the mixture covered over in a small crucible, with about three centner of chloride of sodium, the crucible furnished with a cover, and the assay heated for half an hour in the muffle, or three-quarters of an hour in the wind furnace. The earths are thus slagged off, and a cupriferous matt results in the shape of a button, which is finely pulverised and roasted.

### 2. *The Solvent and Reducing Fusion.*

If the roasted assay, which, besides a small quantity of antimoniates and arseniates, may contain the oxides of copper, lead, iron, zinc, &c., is subjected to a reduction smelting with simultaneous use of solvent agents (borax, glass), then, by a suitable and not too high a temperature, the more difficultly reducible oxides of iron, manganese, zinc, &c., are in great part slagged off, while the oxide of copper is reduced, together with a small portion of the above oxides and most of the oxide of lead, and yields a button of impure copper (black copper), in which also is found almost the whole of the antimony and arsenic of the roasted ore. If too much solvent flux is used, copper also is slagged, which may be known by the red colour of the slag produced. With a lack of solvent agents, a great part of the foreign oxides is reduced, and a very impure black copper is formed, whose refining is attended with greater loss. The charging has been well chosen, when with a black or bottle-green slag a malleable button with a copper-red fracture is produced. The presence of much lead occasions a slagging of copper, while iron, on the other hand, protects the copper. Arsenic and antimony aid in the collection of the copper, since the black copper is thereby rendered more fusible.

The fluxes used in the smelting must be free from sulphur. With an ore containing less than forty per cent. of copper, it may be made with fourteen argol and eight saltpetre; with forty to fifty per cent. of copper, with sixteen argol and eight saltpetre; and with fifty to seventy per cent. of copper, with twenty argol, and eight saltpetre. The more saltpetre is present the more does the copper incline to slag.

Instead of the black flux—if this contains sulphur—a mixture of one hundred parts of pure carbonate of potassa, and ten to twelve parts of flour, is used.

If black flux is used, the charge for one centner of ore consists of two and a half to three centner of black flux, twenty-five to fifty pounds of borax-glass, and fifty pounds or less of glass that is free from lead and arsenic. The ore is rubbed together in a porcelain or serpentine mortar with one-third of the black flux, placed quickly in a crucible, the other two-thirds of black flux added; twenty-five pounds of borax, and thirty to fifty pounds of glass spread over it, the whole covered over with two to three centner of chloride of sodium, and on the top is laid a piece of coal about the size of a half inch cube. The upper layer of black flux prevents the ore from being thrown out of contact with it by the foaming up of the mass consequent upon the reduction. If the piece of coal is taken too large, almost all the chloride of sodium soaks into it, and the assay is too much denuded of slag. In the absence of coal, copper is apt to slag. Difficultly fusible ores require a smaller addition of glass, as this itself is rather difficultly fusible. For the better collection of the copper, with richer ores, an addition of five to fifteen per cent. of arsenic is often given. If the ores contain sufficient lead, no addition of arsenic is required. An addition of as high as ten per cent. of iron, with ores that are poor in iron, is very useful both in the reduction smelting and in the subsequent refining of the black copper.

The presence of protoxide of iron is the safest means to obviate a slagging of the copper, and seems to prevent it to a greater extent, and with more certainty, than a change in the ratio of the saltpetre to the argol in the preparation of the black flux, or than the use of a lower and less prolonged temperature in the smelting. Since protoxide of iron is frequently already present in the roasted assay, an addition of oxide of iron in the smelting is not always necessary; but the more the proportion of copper in the assay sample increases, the more useful does such an addition prove; so that no error is committed if an addition of from one half to an equal weight of pure oxide of iron, or forge scales, is given to every assay in the smelting, or the assay mixed before the roasting with pure pyrites. The latter diminishes also a loss of silver in the roasting.

Reiterated experience has shown that oxide of iron prevents the slagging of copper, and, particularly by Wehrle, an addition of the same is recommended with substances rich in quartz. Dr. W. Fuchs has also drawn attention to this, and by his experiments is led to the conclusion, that a weight of protoxide of iron equal to that of the black flux, can unite with the potash of the black flux to a chemical compound of the formula 2FeO,KO, and that if so much oxide of iron is added to the assay that FeO,KO is formed, the copper is made secure from slagging; further, that by the addition of oxide of iron, the black flux (best made from two saltpetre and five argol) is rendered more fusible.

Since it has long been known that fusing alkaline carbonate is decomposed by oxide of copper, it must be admitted that oxide of iron can separate oxide of copper from its combination with alkali, though this separation may not perhaps be wholly complete.

The assays are exposed to a yellowish-white heat for half an hour to an hour in the muffle or wind furnace (in the Unterhartz, ore and matt assays are allowed to remain in the wind furnace thirteen minutes, and slag assays a quarter of an hour after the fire is well ignited), and when the muffle furnace is used, glowing coals are laid before the crucibles about half-way up to their tops. The contents of the crucibles must be completely fused.

With an assay that has succeeded well (that is, with proper charging and temperature), neither the salt covering nor the slag is reddened with suboxide of copper. The slag is blackish-green from protoxide of iron, glassy, uniform, and easily snaps in pieces. With a red, and therefore cupriferous slag, either the temperature was too high or too long continued, or too little coal, or too much borax and glass present, or the assay too difficultly fusible, which last may be known from the appearance of the heterogeneous, porous slag. Unburned coal must remain on the salt, and at the bottom of the crucible must be found a well fused button of a red or more greyish colour, according to its purity. If there is found between the copper button and the slag a brittle crust or layer of matt, the roasting was not complete, and the assay is to be thrown away.

Only when substances are to be examined which contain nothing but sulphides of iron and copper, and besides have but little or no earthy gangue, provided too that the quantity of copper is not very small, can a copper button be obtained by roasting and reduction smelting with a well-proportioned mixture, whose weight shall give the contents of the ore with sufficient accuracy. But the metallic button obtained must then, with a slag that is free from copper, have all the characteristics of pure copper, must upon its surface, as well as in the fracture, be pure copper red, and be capable of being hammered without breaking or cracking.

If the colour and malleability of the copper button prove that the impurities cannot amount to over one to three per cent. of the weight of the copper, a further refining is omitted on account of the loss thereby occurring. (Mansfeld copper matts.)

All metallic oxides still present in the roasted assay sample, which are as easily reduced as oxide of copper, and whose metals are fusible at the temperature used, either by themselves or when alloyed with copper, pass into the copper as it separates out. In all cases, therefore, when the roasted assay still contains such metals, among which are lead, bismuth, tin, cobalt, nickel, antimony, arsenic, &c., there can be no pure copper produced, and the copper then obtained is designated by the name of *black* copper. This black copper must then, by a third operation, be freed from these foreign ingredients, to which, if the roasting was not exceedingly thorough, sulphur may also be added

### 3. *The Refining of the Copper on the Cupel or on the Refining Dish.*

This operation aims at the removal of the foreign ingredients from the black copper.

For this purpose the copper is brought to fusion, and access of air allowed. Thus the constituents of the black copper, which are more easily oxidised than copper, namely, phosphorus, sulphur, arsenic, antimony, lead, iron, bismuth, zinc, &c., are next converted into oxides, and may be removed as such. A partial oxidation of the copper at the same time, however, cannot be entirely avoided here; and, moreover, when it is attempted to prevent too great an oxidation of the copper, considerable traces of the substances named above are apt to remain behind in the refined copper. From other metals, namely, from gold, silver, nickel, cobalt, &c., the copper cannot be freed at all, or only imperfectly so, by this method.

This is the reason of the imperfection of this mode of refining, and, therefore, of all methods of assaying which involve it. The most practised assayer, with all his skill and experience, cannot entirely remove this imperfection. This assay, however, none the less deserves to be used, for with acquired practice it yields a result in a *shorter time*, though it be but more or less approximately correct, than it is possible to obtain one in the wet way. Moreover, rightly conducted assays, compared by the differences usually occurring between them, remain always, or nearly so, quite as reliable as many other metal assays in the dry way, *e.g.* the lead assay.

The assay is in general considered as successful with rich and medium ores, when with correct management the weight of two duplicate assays does not differ by more than one per cent.

This refining is performed in different ways.

*a. Refining upon the refining dish.*—This method is the one most frequently chosen in Germany, *e.g.* at Freiberg, and at the Victor Frederic smelting-house, and also in Hungary. It is especially applicable when the black copper does not contain very much lead, and is the more reliable the purer the black copper already is.

By this method indeed an oxidation of the copper cannot be wholly prevented, but under favourable circumstances the loss thus occurring is vanishingly small. In Freiberg the copper button, with as little borax as possible, generally an equal weight, wrapped in a cornet of letter-paper, is placed on the very flat, white-hot dish, surrounded by glowing coals, and fused quickly at as high a temperature as possible. A slow fusion occasions oxidation and slagging of the copper. If black copper, which contains neither arsenic, antimony, nor lead, is to be examined, then to a fifty pound assay, five to ten per cent. of lead and thirty to fifty per cent. of borax, as may be required, are added, in which case the borax is generally fused first on the white-hot dish, then the copper, and afterwards the lead added. If the copper to be refined is not in a single piece, then, in order to avoid loss, it is never placed on the dish at the same time with the borax, but only after the intumescence of the latter has ceased. A black copper containing sulphur sparkles when placed on the dish. Cornets of weighed lead and borax must always be at hand to add in case of necessity.

As soon as the copper shows a convex, perfectly clear surface, and is surrounded by a thinly fluid ring of borax slag, the mouth of the muffle is slightly opened to give access to the oxygen necessary for the oxidation. If the copper is not clear, but covered with a black crust, while at the same time the muffle is white hot, the operator tries adding borax. If this alone does not help the matter, a cornet of lead is added, and the heat increased, if possible, when the black copper soon presents a clear surface. Very impure black copper with only forty to fifty per cent. of copper, is placed on the dish at first with a large quantity of borax only, without lead, and the latter added only when the copper is sufficiently refined. Special care must now be taken that the temperature does not sink too low. If *lead* is present fumes of it rise at this period. A portion of the lead goes into the slag. *Arsenic* mostly passes off in fumes; a portion of it, however, remains in the slag as arseniate of iron. *Antimony* behaves similarly, only it is more obstinately retained by the copper. *Nickel* is the most difficult to scorify, and can only be slagged by a large addition of lead, and in consequence of this, with a considerable loss of copper. If too little borax is present the slag is apt to become stiff, or solidify.

When the copper is nearly refined, it 'brightens' like silver, only less distinctly; the 'brightening' is particularly to be seen at the lower edge of the metal. In the presence of antimony and arsenic, the 'brightening' is less distinct than with lead, but it becomes so also with the latter when the buttons are small. In the latter case the assay is assumed to be done, when it no longer fumes. The temperature must, at the instant of 'brightening,' be exactly at the point at which the copper solidifies, since otherwise it would continue to oxidise; but good care must be taken that the copper does not solidify too soon. It shows in the 'brightening' a peculiar green colour. The assay is now removed from the furnace, carefully quenched in water, and freed from the slag.

The buttons should not differ by more than one to two per cent. in weight, and the value is stated only in whole pounds through all degrees of richness.

In a good assay the button has the pure copper colour, is ductile, and uniformly granular and rose-red in the fracture. A button not sufficiently refined is red exteriorly, but the fracture is grey; an over-refined one, dark red exteriorly and brittle, the fracture more smooth than granular, and with a high over-refining, even laminated, moreover, the slag is then red. With proper refining, the slag is blackish-green, from the presence of iron. If lead were present, the slag is greenish-blue at the edges from iron, nearer the button it is yellowish-red from $PbO,Cu_2O$, and at the button itself suboxide of copper appears. The yellowish-red colour must not be confounded with that of the basic arseniate of iron, which forms copper-red spangles on the surface of a slag that is saturated with it. Since the adding of lead involves an unavoidable loss of copper, an addition is necessary to the amount of copper found, in order to learn the correct contents of the black copper. Empirically, to every ten pounds of metal slagged off (from the black copper and the lead added) one pound of copper is reckoned as also slagged. In many localities, for every five pounds of loss (of the black copper) one pound more of copper is reckoned. If there is a lack of borax in the refining, more may be added in the process. If the dish then becomes too full, it is cooled in water, the copper freed from the slag, and again mixed with borax on a new dish. But then a loss of copper is more apt to occur, as most of the iron, which otherwise protects the copper from slagging, is already slagged off. It is therefore sought in preference to get through with a single operation.

If much arseniate of iron forms, which begins to make the slag stiff, no more borax can be added, as the assay is thereby completely chilled and cannot be again rendered fluid. In such a case the assay must be freed from the slag. A new addition of borax, therefore, should only be given in the commencement, while the slag is yet entirely fluid and the button is not yet clear. When the button once becomes clear, a further addition of borax is seldom necessary if the furnace is kept hot enough. Only very impure black coppers do not then refine with a single operation.

Plumbiferous black copper is already copper-coloured, has a certain softness, and quickly refines. Arsenical black copper refines more slowly, requires more borax on account of the iron it contains, and never gives a refined copper of a fine red colour. So long as it is not yet refined, if the heat is high enough, it is movable on the dish. With very arsenical copper, if the heat is very strong, a blue arsenic flame bursts forth, which lifts the button. A constant blue halo of burning arsenic is often seen.

Gold and silver remain in the copper, and must be taken account of, when they amount to one-half per cent. Much silver makes the copper white. Generally, only two assays are carried on at once; however, with proper attention, four assays may be made at the same time.

*b. Refining on the cupel.*—This method has been in use at different places, *e.g.* at the Oberhartz and Unterhartz smelting works, since the time of Schlüter. It is by no means more accurate than the above, but is, perhaps, the most suitable one for very plumbiferous black copper.

A quantity of pure copper equal in weight to the black copper is weighed out, while two cupels are brought to a white heat in the muffle of the assay furnace. Upon each of the cupels an equal weight of pure lead is placed, and when this has begun to 'drive,' the black copper is placed on one cupel and the pure copper on the other, whereupon the muffle is again closed till the alloy on the cupel once more 'drives' well. Sometimes (as at the Unterhartz) half of the lead is placed on the cupel with the copper, and as soon as this is red-hot, the other half of the lead added. The quantity of lead to be used depends upon the nature of the black copper; if this is very plumbiferous, an equal weight of lead may suffice; if the black copper is almost or entirely free from lead, and is also impure, two and a half to four parts by weight of lead must be added (at the Unterhartz, for example, four parts of lead are taken). During the 'driving,' in which the muffle is fully one half opened, without however closing the draught of the furnace, a sufficient heat is to be secured, and it must be especially seen to that both cupels remain equally hot, so that the purification of the copper may be as nearly equal as possible on the two. The higher the heat in the 'driving,' the purer does the copper become, and the rounder is its form. The 'brightening' of the assay is somewhat more distinct than in the refining on the refining dish. As soon as it has ensued, a spoonful of coal-dust or borax is poured over the cupels in the muffle. They are then immediately taken out of the furnace and cooled in water. The indications of the refinement of the copper are the same as above; it is difficult, however, to obtain buttons wholly free from lead. The simultaneous and similarly conducted cupellation of a quantity of pure copper equal in weight to the black copper, with the assay, is intended to make a calculation possible of the quantity of copper, which the black copper has lost by scorification. What the pure copper has lost in weight is added to the weight of refined copper obtained from the black copper. An example will make this clearer. Let the black copper weigh seventy-five pounds. Seventy-five pounds then of pure copper may be cupelled with $75 \times 3 = 225$ pounds of pure lead, and sixty pounds of copper be recovered from it, so that the copper consumed has amounted to $75 - 60 = 15$ pounds. Let the black copper, likewise cupelled with two hundred and twenty-five pounds of lead, have yielded forty pounds of refined copper; then the quantity of copper contained in the seventy-five pounds of black copper amounts to $40 + 15 = 55$ pounds, which is the amount to be stated, if the black copper did not contain lead. If, however, the black copper is very rich in lead, a second correction is also made. The difference, $75 - 55 = 20$ pounds, is considered as lead. Now, as in the assay with pure copper, two hundred and twenty-five pounds of lead have slagged fifteen pounds of copper; these twenty pounds of lead are assumed to have slagged $\frac{20 \times 15}{225} = 1\frac{1}{3}$ pounds more of copper. The quantity of copper to be reported, therefore, amounts to $40 + 15 + 1\frac{1}{3} = 56\frac{1}{3}$ pounds.

If the black copper is so much contaminated with lead that by refining in the manner specified no copper would be obtained from it, an equal weight of pure copper is added to it, and a double weight of pure copper simultaneously cupelled upon the second cupel. For example, let the black copper weigh seventy-five pounds; then seventy-five pounds of pure copper are added to it, and cupelled with 2 × 75 × 3 = 450 pounds of lead. On the other cupel, 75 × 2 = 150 pounds of pure copper are cupelled with four hundred and fifty pounds of lead. The calculation is made as above, only the seventy-five pounds of copper added are finally again deducted. If the quantity of lead to be added is diminished, this must be done equally in the actual assay and the controlling one.

## *b. Assays of Poor Ores and Products of Class I.*

### 1. CONCENTRATION FUSION.

Five to ten centner and more of the unroasted ore are mixed with fifteen to twenty per cent. of iron pyrites free from copper, in case pyrites is not already present in the ore, and the assay smelted with an addition of fifty to one hundred per cent. of borax, under a thick layer of chloride of sodium in a clay crucible at an incipient white heat. By this process a button of crude matt is obtained, in which the copper of the assay sample is found concentrated as sulphide of copper. This crude matt is weighed, and then first subjected to a copper assay by roasting, reduction smelting, &c. If several assay centner have been taken for the concentration smelting, the final proportion of copper found is calculated accordingly.

A slagging of copper is indeed seldom entirely avoided by this concentration smelting, but the loss of copper is generally less than if such poor ores were at once, without any previous work, subjected to the ordinary process in the dry way, which would sometimes yield no button of copper at all, since the little copper in the ore is lost in the slag.

According to Fuchs, for the most complete prevention possible of a loss of metal, and for the purifying of the copper, the well roasted ore is placed in an assay crucible while still hot, with twenty per cent. of pyrites and twenty per cent. of sulphur, covered with powdered glass and some vitrified borax, and smelted to matt.

### 2. Fusion with Collecting Agents.

With *poor* and *impure* ores, if the ordinary method is used, errors are very apt to arise, from the infusibility of the ore and from the basic nature of the suboxide of copper, which, especially in the presence of silica, is very much inclined to slag. In such cases, as practised in Freiberg, lead, litharge, arsenic, antimony, arsenious acid, or oxide of antimony, is added for the collection of the copper while smelting to black copper. These fluxes have their advantages but also their disadvantages. Five to fifteen per cent. of *lead* or *litharge* gives with copper an easily fusible alloy, which collects better together, but also carries copper into the slag, which consequently becomes red. If the result of experience is taken as a basis, that, in the refining smelting, ten pounds of lead slag one pound of copper, then if five per cent. of lead are added to an ore with one per cent. of copper, only half a pound of copper is obtained, and with thirty per cent. of lead no copper at all would be obtained. The intimate mixture of the lead with the ore has also its difficulties, and therefore it is better in this connection to use litharge.

By an addition of *arsenic*, *antimony*, *arsenious acid*, or *oxide of antimony*, easily fusible compounds of arsenic and antimony with copper are obtained, the mixing can be accomplished more thoroughly than with lead, and for poor ores, such an addition is more suitable than one of lead. On account of danger in dealing with arsenious acid, it is best to use metallic arsenic, which does less harm in the refining than antimony. In this operation, indeed, neither of the two is entirely removed from the copper, and they impart some brittleness to it, and a greyish colour to the fracture. The assayer may, however, be satisfied with the result of an assay treated with arsenic

### *c. Assays of Oxidised Ores and Products of Class II.*

Oxidised copper ores and products are—

1. Without previous roasting, subjected to a solvent and reducing fusion, and the black copper thus produced, refined, if necessary (richer ores and products, *e.g.* red copper, malachite, refinery slags, &c.).

2. After previous roasting, smelted to black copper, (*e.g.* sulphate of copper, copper ores which contain arsenic acid, antimonic acid, &c., ores imperfectly roasted in the large way, many copper ore slags, cement copper slimes, which may contain basic sulphate and arsenate of iron, &c.) The roasting is performed with an addition of coal-dust or graphite, and finished, if necessary, with carbonate of ammonia.

3. Smelted to black copper with collecting agents (lead or arsenic). Poor oxidised copper ores, especially the quartzose ones, must always be treated in this way. For example, poor copper ore with chalybeate and basic gangue without silica is mixed with ten per cent. of arsenic, thirty to forty per cent. of borax, and twenty to twenty-five per cent. of glass; if it contains pyrites, mispickel, quartz, and calcspar, with twelve per cent. of arsenic, thirty per cent. of borax, and thirty per cent. of glass, besides black flux; ferruginous, quartzose malachite, with ten per cent. of arsenic, sixty per cent. of borax, and fifteen per cent. of glass. If poor oxidised ores are free from iron, ten to twenty per cent. of oxide of iron is added. Also richer oxidised substances are advantageously mixed with collecting agents, *e.g.* refining slag, with five per cent. of arsenic, thirty per cent. of borax, and thirty per cent. of glass.

4. Subjected to a concentration fusion, with twenty to twenty-five per cent. of pyrites free from copper and twenty per cent. of sulphur, one hundred per cent. of vitrified borax, one hundred per cent. of glass, and twenty to twenty-five per cent. of colophony with a covering of salt, and the resulting matt treated like a sulphuretted ore. The ore is placed on top of the pyrites and sulphur, and covered over with the fluxes, &c.

### *d. Copper Alloys. Class III.*

But very few copper alloys can be refined by the above process. For the *black copper* produced by the copper smelting process, everything is observed which was prescribed for the refining of the buttons resulting from the reduction smelting. If the refining is to be done on the cupel, one or more rarely two centner are weighed out for the assay. *Cupriferous lead* is refined on the cupel. Two centner of the cupriferous lead and half a centner of pure copper are placed on one cupel, and on the other, two centner of pure lead, and half a centner of pure copper, and the two are cupelled at an equal heat. If now, for example, thirty-six pounds of copper have been obtained on the first cupel, and twenty-seven pounds on the second, then in the two centner of the assay substance there were contained $36 - 27 = 9$ pounds, or four and a half per cent. of copper.

Heine gives for the examination of the Mansfeld *black copper*, which seldom contains more foreign ingredients than four to seven per cent. of iron, a little sulphur, and only traces of zinc, cobalt, nickel, lead, and phosphorus (the whole amount of the substances last named is not generally over one per cent.), the following directions :—

One or half an assay centner of the black copper in filings is weighed out in duplicate, and placed in the muffle, at first with a gentle, then with a stronger heat, and with frequent rubbing up, until all the copper appears black and changed to oxide, and no more grains can be felt with the pestle. The roasted assay is now mixed with two to three centner of black flux and one to one and a half centner of glass, free from lead and arsenic, the charge placed in a crucible for assaying copper, covered with salt, and smelted in the wind furnace. The black flux for this assay must consist of at least twenty parts of argol to eight parts of saltpetre, since with a smaller quantity of argol red slags are generally produced. Should this still be the case, six to eight assay pounds of coal-dust are added to the charge. Heine obtained in this way a copper, which he considered quite as well refined as that produced by the preceding processes.

These directions may, perhaps, deserve to be followed with black copper similar to this, as they allow us to dispense with the always imperfect refining on the cupel or refining dish; especially if, for reasons already explained, a portion of pure oxide of iron or forge scales is mixed with the roasted assay in the smelting, in order the more certainly to avoid the formation of a red slag, which always indicates loss.

The most frequent alloys of copper, i.e. *brass*, *German silver*, *gun metal*, &c., cannot be assayed in a reliable manner in the dry way. German silver, because the nickel could not be removed at all, or only with great difficulty, and the rest because zinc and tin give such difficultly fusible oxides, that they could not be properly removed in the refining.

In the *alloys of copper with silver*, *gold*, and *platinum*, the copper may be determined from the loss arising from cupellation with lead.

### *Remarks upon the Copper Assays in the Dry Way.*

These assays are burdened with various defects. The roasting is an exceedingly tedious process, and only by a gradually increasing temperature, by repeated grinding up and mixing of the roasting substance with coal-dust, graphite, or carbonate of ammonia, is it possible to sufficiently remove the sulphur. In the reduction smelting, loss of copper is apt to occur, if a correct temperature and a suitable charging are not employed. If the fluxes (borax, glass, &c.) are present in too large quantity, copper is slagged; if there is a lack of them, a very impure black copper is produced, with whose diminishing richness in copper, the loss of copper in the refining increases. The last-mentioned operation is in and of itself imperfect, and on account of the high temperature it requires, and the ever necessary attention of the assayer, very troublesome.

The poorer the substance is in copper, the more unreliable do the results of the assays become.

In all docimastic assays of copper in the dry way, the *silver* or *auriferous silver* contained in the assay sample cannot be removed, and it has generally pretty completely collected in the copper obtained. These copper assays give nowhere any indications whether gold or silver is present or not; and the amount of these metals which may be present must therefore be both sought for and determined by a special assay for them. If they are found, and in sufficiently large quantity, they are deducted from the weight of the copper.

The dry assay is mostly found in practice in smelting works, where even in the hands of less scientifically educated than skilful assayers, with the character of the assay substance once known, and suitable practice in following out the separate manipulations, it gives results which suffice for the business of working copper in the large way.

## B. ASSAYS IN THE WET WAY.

### I. *For Substances rich in Copper.*

### *a.* KERL'S MODIFIED SWEDISH ASSAY.

One assay centner of finely rubbed ore, &c., is warmed in an assay flask or beaker, on the sand-bath, with as little as possible of aqua regia (two parts crude hydrochloric, and one part crude nitric acid), and as soon as the proper decomposition of the assay substance has taken place, in order to expel the nitric acid, evaporated nearly to dryness with a few drops of oil of vitriol. In the presence of nitric acid the copper would be only imperfectly precipitated by iron; the presence of hydrochloric acid does no harm. Also the nitric acid may be destroyed by heating the solution containing it with crystals of protosulphate of iron, only the work then passes off less cleanly than if sulphuric acid is used.

The still damp mass is cautiously moistened with hot water, and filtered into a beaker, the residue no longer containing any particles of ore, &c., is washed out a few times with boiling water (till a drop of the washings no longer deposits a brownish coating of copper on a clean iron wire), the filtrate, about 150 to 160 cubic centimetres, heated nearly to boiling with a few pieces of iron wire about two inches long and two lines thick, till a brownish coating no longer forms on a clean iron wire held in the solution. The approximation to this point is indicated by the solution's becoming colourless. Should the mass become dry in the evaporation with sulphuric acid, it is moistened before filtering with a few drops of sulphuric acid, in order to make the basic salts formed soluble. The concentrated solution is not filtered on to the pieces of iron wire, but these are placed in the solution after it is diluted with the washings, because otherwise they become so rapidly and so firmly coated over with copper, that the latter can only with difficulty be separated from them. The pieces of wire are so taken as to correspond in size with the beaker used, and of such a length as to cross one another in it, in order to present as much surface as possible to the direct contact of the fluid.

After the precipitation is ended, the beaker is poured full of hot water, which, after some time, is decanted, care being taken that no copper goes with it, the beaker again filled with hot water, a porcelain saucer placed bottom upwards on the top of it, and beaker and saucer then inverted so that the iron wires, together with the copper and some of the fluid, sink into the saucer. When this has taken place completely, the beaker is removed from the saucer by drawing it quickly over the side with the hand, the iron wires freed by rubbing them with the fingers from the copper coating, which does not adhere firmly, well rinsed off, and the copper well washed two or three times by decantation with hot water. The decanted fluid may be placed in a beaker, and allowed to stand quiet for some time, that it may again deposit any very finely divided copper suspended in it. The latter is not to be confounded with the particles of carbon separated from the iron wires, and which pass off in part in the decanting. Owing to the short time occupied by the precipitation of the copper, few or no particles of carbon and iron are detached from the wires, as in the older assays, and the copper is precipitated with a pure metallic colour, without being in the least contaminated by basic salts of iron.

The damp copper, freed from water as much as possible (if necessary, by removing it with blotting-paper), is dried at a gentle heat in an atmosphere freed from acid vapours until two successive weighings give the same result. An oxidation of the damp copper may be counteracted by adding alcohol to it, and also by covering over the saucer. Overmuch care in the drying is not required, for even at a somewhat higher temperature the increase in weight is but very small, so long as the copper retains its proper colour. The precipitated copper should not be allowed to remain too long in contact with free sulphuric acid, since it is oxidised by it.

This process alone can only be used when in the substance to be examined there are no other metals present (*tin*, *antimony*, *arsenic*, *gold*, *bismuth*) which are likewise thrown down by iron. *Silver*, *lead*, and *mercury* are indeed also precipitated by iron, but these metals can be readily removed. *Silver* and *lead* remain in the insoluble residue, the first as chloride of silver, and the last as sulphate of lead, and may be determined by assaying it after incinerating the filter. *Mercury* (contained, for example, in many fahlerzen) is indeed thrown down with the copper, but is volatilised by igniting the copper in a porcelain crucible or on a roasting dish in the muffle, whereby the copper also passes into pulverulent black oxide, and after two ignitions is weighed as such. One hundred parts of oxide of copper contain 79·826 of metallic copper. The oxidation may be promoted by moistening the copper with nitric acid before ignition.

The so-called *copper slate* (Kupferschiefer) must, before treating with aqua regia, be ignited to remove the bitumen. If the substance cannot be completely decomposed by aqua regia (many *slags*, for example), it is first fused in the finely pulverised state, with about two to two and a half parts of carbonate of potassa, or calcined carbonate of soda, in a clay crucible at a red heat in the muffle or in a platinum crucible over a spirit-lamp. If the assay sample contains *lime*, difficultly soluble sulphate of lime is formed, which may, however, be dissolved out by repeatedly washing the copper with boiling water.

The presence of *iron*, *nickel*, *cobalt*, *manganese*, and *zinc* in the assay substance does no harm, as their sulphates are not decomposed by iron.

While by the older method an assay might last from one to two days, during which time, indeed, as many assays might be simultaneously conducted as the arrangements otherwise allowed, all the operations of this newer method require but from two and a half to three hours, with an ore containing two to seventy per cent. and more of copper.

With respect to the accuracy of the assay, with suitable substances, as the experiments at the Oberhartz smelting-house and the investigations of Von Hubert have proved, it leaves nothing more to be desired as a business assay. At the smelting-house alluded to, the different assayers are allowed two per cent. difference, which is, however, very large.

Mohr has given an analytical accuracy to this assay, by performing all the operations as much as possible in the same vessels, conducting the washing with care, and precipitating the copper with granulated zinc free from carbon, instead of using iron, which liberates coal. As a business assay, however, the Oberhartz method is to be preferred on account of its simpler practicability.

If *bismuth*, *gold*, *tin*, *antimony*, or *arsenic* are present in the substance to be assayed, the method described requires different modifications according to whether *arsenic* is present or not. The presence of *bismuth*, which, moreover, seldom occurs (*e.g.* in many *fahlerzen*, *cupreous bismuth*), also necessitates a special assay treatment.

*a. Arsenic is not present.* One assay centner of the ore, &c., is decomposed at a more or less high temperature by nitric acid, so that *gold*, *oxide of antimony*, and *oxide of tin*, remain in the residue, as also *silver*, if some chloride of sodium is added to the solution. It is now filtered, the precipitate washed with water, the filtrate evaporated, to expel the nitric acid, and to separate any *lead* that may be present, with sulphuric acid, and the copper precipitated from the filtrate in the usual way. Mercury, if present, is removed in the manner stated above.

*b. Arsenic is present.* One assay centner of ore is decomposed in a beaker or a roomy flask with as little aqua regia as possible, the free acid neutralised with soda, or even supersaturated so that a precipitate separates, and the mass digested with excess of solution of sulphide of sodium for some time (perhaps half to three quarters of an hour) at almost boiling-heat. The solution of sulphide of sodium is prepared by igniting and lixiviating a mixture of two parts of anhydrous carbonate of soda and one part of coal-dust or flour. To the filtered solution of this salt flowers of sulphur are added in excess, which partially dissolve in it and increase its capacity for dissolving the electro-negative sulphides of *gold*, *antimony*, *arsenic*, and *tin*, by forming with them sulphur salts, while *silver*, *lead*, *copper*, *mercury*, *iron*, *zinc*, *manganese*, *nickel*, and *cobalt* are sulphurised by the solution, but not dissolved. The two groups of metallic sulphides are separated by filtering, and the sulphides of the last group, which remain on the filter, well washed with cold water. The finger is then held over the bottom of the funnel, concentrated nitric acid poured on the filter, and the sides of the funnel carefully heated by slowly revolving it over a spirit-lamp. The sulphides thus partially dissolve in the acid, but separate completely from the filter, so that by punching a hole through the bottom of it they can without difficulty be washed into a beaker. Here they are completely decomposed by heating with the nitric acid, the latter removed with simultaneous separation of the lead, by heating with sulphuric acid, and the process continued as heretofore prescribed.

*c. Bismuth is present.* After *gold*, *antimony*, *arsenic*, and *tin* have been removed, if necessary, in the manner prescribed under *a* or *b*, carbonate of ammonia in excess is added to the solution obtained, which contains copper and bismuth. By this the copper is dissolved, while *bismuth*, *lead*, and *mercury* are precipitated as carbonates. After a while the blue copper solution is filtered off, the carbonate of ammonia supersaturated with sulphuric acid, and the copper precipitated from the solution with iron. Should much iron and alumina be contained in the solution that has been freed from gold, antimony, arsenic, and tin, then in the precipitation with carbonate of ammonia a slimy precipitate is formed which may retain much copper. In such a case the solution is evaporated with sulphuric acid, and the copper precipitated in the usual way with iron. Since, however, the copper then contains bismuth and mercury, it must be redissolved in nitric acid, and the solution treated as above with carbonate of ammonia.

Synthetic experiments with substances of most varied composition have proved that this modified assay yields sufficiently correct results as a docimastic assay with ores containing two to seventy per cent. and more of copper. Either the precise percentage of copper weighed out is again obtained, or with the richer ores a difference of at most two per cent. occurs between several assays of one and the same kind.

By means of the Oberhartz assay and Heine's colorimetric assay the copper contents of all substances, rich and poor, can be docimastically determined with sufficient accuracy. By the Oberhartz assay, proportions of two to three per cent. and over are determined, and by Heine's mode the lesser ones down to ·03 per cent.

### *b.* ASSAY OF COPPER BY PRECIPITATION WITH METALLIC ZINC.

The precipitation of copper from its solutions is of old date. Fresenius recommends the process, the accuracy of which he has tested, in his 'Quantitative Chemical Analysis.' The process is performed in the following manner. The solution, either in hydrochloric or sulphuric acid (nitric acid, if present, must be expelled by evaporation with sulphuric acid), is transferred to a weighed platinum dish, diluted so as to ensure a moderate and steady evolution of gas, and a piece of pure zinc introduced. The dish is covered with a watch-glass, which is afterwards rinsed into the dish. Heat promotes the reaction, but is not strictly necessary. The copper is deposited partly as a coating upon the platinum, partly in the form of spongy masses. Sufficient acid must be always present to maintain the evolution of hydrogen. In about an hour or two, all the copper will have separated: a few drops of the solution must be tested with sulphuretted hydrogen water to make this certain. If no colour has been imparted to the solution, it may be assumed that all the copper has been deposited. The metal in the dish is now examined for undissolved zinc by pressing with a glass rod, and by addition of hydrochloric acid. All the zinc being dissolved, the copper is pressed together with the glass rod, the supernatant fluid decanted, and hot water poured into the dish and repeatedly decanted, without loss of time. The washing is continued until the decanted fluid is quite free from hydrochloric acid. When such is the case, the water is decanted, as far as practicable, rinsed with alcohol, and dried upon the water-bath: as soon as the dish is cool, it is weighed. The precipitation can be effected in a porcelain or glass dish, though the process will occupy more time, and the copper will be deposited in non-adherent spongy masses.

### *c.* COLORIMETRIC COPPER ASSAYS.

These are based upon the fact that ammonia added in excess to the solutions of salts of copper, produces a beautiful azure blue colour, whose intensity depends upon the quantity of copper dissolved. By comparing the shades of blue colour in equally thick layers of the dissolved ammoniated assay substance (assay fluid) with a normal or standard ammoniated fluid whose copper contents are known, the quantity of copper in the former can be calculated when its volume is measured.

To Heine, the superintendent of the smelting works in Mansfeld, belongs the merit of having first successfully employed this reaction for the determination of small percentages of copper, and later it has been also extended by Jacquelain, Von Hubert, and Müller, to the determination of larger quantities of copper.

### 1. Heine's Colorimetric Method.

For the docimastic determination of the quantity of copper in bodies poor in this metal, *e.g.* in slags, lead matte, litharge, crude lead, and other plumbiferous metallurgical products, tin, cupelled silver, &c.; in short, in all substances which contain from a trace to about one per cent. or a little more of copper, this method is the most advantageous to be used.

After the assay sample has been reduced to as fine a state of mechanical subdivision as possible, which with slags is best attained by sifting or washing them, one centner (3–4 grammes) of it is weighed out and dissolved or so fully decomposed by a suitable acid that in the residue, which is to be filtered and well washed, no more copper remains behind. For this purpose nitric acid or aqua regia is employed, according to the character and peculiar behaviour of the substance, and the nitric acid is concentrated or somewhat diluted, as may be required. The solution is either immediately, or after the copper has been first precipitated by hydro-sulphuric acid gas or iron and again dissolved, strongly supersaturated with caustic ammonia, and the precipitate, if any, thereby produced, steeped in the caustic ammonia for a considerable time, and with frequent stirring at a very gentle heat (30°–40° C.), then filtered off and thoroughly washed. According to the quantity of copper present, and according to the degree of dilution, will the solution obtained, which if it should become at all turbid must be once more quickly filtered (*e.g.* with refined and crude lead), appear more or less strongly coloured blue. The volume of the solution is measured in graduated vessels, and the intensity of the colour compared with and determined from fluids, which have been previously prepared as standard fluids, and which, for a definite volume, contain a definite, accurately weighed quantity of copper, that has been dissolved in nitric acid, precipitated by caustic ammonia, and redissolved in excess of the same. From the measured volume, and the intensity found by comparison, the quantity of copper is then determined by calculation.

Heine proposes standard fluids with one, two, three, and four assay loth of copper in one ounce (two loth, commercial weight) of the ammoniated fluid. These four standard fluids are all-sufficient.

If the French weights and measures are used, standard fluids are taken with ·001 ·002 ·003 ·004 grammes of copper to every twenty-five cubic centimetres of the fluid.

The graduated vessels (cylinders) required for the preparation of the standard fluids, as well as for the measuring of the assay fluid, can be easily prepared by the assayer himself. One quarter of an ounce of water is weighed out a number of times in succession and poured into the cylinder, and each time the height of the fluid is marked in a durable manner on the glass with a diamond, or by etching it with hydrofluoric acid vapours, etc. Also earthen or porcelain measures, that are prepared and marked for the volumes that hold one, two, three, four, &c., ounces of water, may be used.

It is not practicable to replace the volumetric measurement by weighing, for the quality and quantity of those substances which are soluble in acids and not precipitated by ammonia, or are again dissolved by it, may vary greatly in the assay.

In the formation of the normal fluids, two assay pounds of chemically pure (galvanic) copper are weighed out on a good balance, dissolved in nitric acid, the solution supersaturated with caustic ammonia, and placed in a graduated cylinder, which is divided to whole, half, and quarter ounce volumes of water, and then water enough is added to bring the fluid to the sixteen ounce mark. The fluid then contains $\frac{64}{16}=4$ loth of copper per ounce. Six ounces of this four loth solution are then taken, two ounces of water added to it, and eight ounces of fluid obtained, with $\frac{24}{8}=3$ loth of copper to one ounce of water. The two loth solution is formed in a similar way by diluting four ounces of the four loth solution to eight ounces; the one loth, by diluting four ounces of the four loth normal fluid to sixteen ounces. In the measuring of the assay fluid it is estimated within one-eight of an ounce, which is sufficiently close. If in the dilutions a mistake is actually made of one-sixteenth of an ounce, the maximum of possibility, the error amounts to about two cubic centimetres, which in a whole mass of fluid of 200—500 cubic centimetres has no influence upon the solution that can be detected with the eye.

The preservation of the standard fluids, as well as the comparison of the blue assay fluids with them, must take place in glass vessels closed with ground glass stoppers. These vessels must have the same form and size, consist of the same colourless glass, and have an equal thickness of glass in the smooth side walls. The last condition is obtained the surest by grinding. This grinding, however, which notably increases the cost of the glasses, is not indispensably necessary if the vessels are carefully formed and blown in a good glass-house. An oblong form is most advantageous for the vessels. They hold about an ounce and a half of fluid, and are about two inches long, two and a half inches high, and one inch wide, with walls about one-eighth of an inch thick.

The glasses are very advantageously formed from an unblemished sheet of plate glass of equal thickness throughout, either by fusing or cementing the sides together and the insertion of a glass or platinum neck. The assayer has in the form of vessel indicated a triple control in the comparison of the assay fluid with the normal solution according as he looks through the fluid in three different directions.

The digestion of the assay sample with acid may take place in any suitable vessel whatever, a glass flask, a beaker covered with a watch-glass, &c., only no thumping and spirting of the fluid should be possible in the process. The nitric acid, &c., must be added little by little. The time required for this may vary greatly. The solution of *cupelled silver*,* *skimmings*, &c., with nitric acid is finished in a short time; on the other hand, in the examination of difficultly decomposible *slags*, with which concentrated nitric acid or aqua regia will always be used, the digestion often requires to be continued in a warm temperature for two to three times twenty-four hours. The mass must be frequently stirred with a glass rod, because many slags decompose rapidly with evolution of heat, form a thick jelly, and deposit a crust on the bottom of the glass. Sub-, singulo-, and bi-silicate slags, mostly decompose readily, higher silicates resist complete decomposition by aqua regia, and then a preliminary solvent ignition or fusion with carbonate of potassa or calcined carbonate of soda; or better, a mixture of both, is necessary, precisely in the manner given in the wet assay of copper. Here also it does no harm if some of the substance of the crucible remains adhering to it.

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* With cupelled silver, after dissolving in nitric acid, the silver may be precipitated with chloride of sodium, the chloride of silver filtered, washed, and the solution then mixed with caustic ammonia.

The decomposition of the slags by acid is complete when in the stirring with a glass rod no more grating can be perceived.

After hot water has been added to the decomposed assay, the residue is collected on a filter, well washed out, without diluting the filtrate too largely, and the copper precipitated from the solution, if necessary, with hydrosulphuric acid gas, especially when a notable quantity of alumina and iron is present, whose slimy precipitates from the immediate precipitation with ammonia always retain copper. This precipitation of the copper has also the advantage that, as cobalt and nickel do not precipitate with it, the colouring effects which they would produce, if present, are removed. Since the sulphide of copper requires for its solution but a few drops of nitric acid, in the succeeding treatment of the solution with ammonia, but a small quantity of ammoniacal salt is formed, and the specific gravity of the coloured fluid varies but very little from that of water and the normal solution. With the increase of the specific gravity of the assay solution, its volume is considerably increased, and therefore it gives too large a measure in the direct precipitation with ammonia. If the precipitation with hydrosulphuric acid gas is completed in four to six hours, the sulphide of copper is filtered out, thoroughly washed with cold water containing hydrosulphuric acid, the filter dried, ignited in a porcelain crucible, the oxide of copper formed, warmed with a few drops of nitric acid or aqua regia, supersaturated with ammonia, filtered, and well washed, till the washings are no longer tinged bluish.

A precipitation of the copper with iron wire, from a solution evaporated with sulphuric acid, and a re-solution of the copper in nitric acid, consumes less time. If the copper is not previously precipitated, errors of some thirty per cent. and more of the whole amount of copper may occur. By repeated solution of the iron precipitate and precipitation with ammonia, all the copper cannot, however, be extracted.

In the examination of *litharge*, the solution in nitric acid may be dispensed with. The oxide of copper can be at once extracted from it with caustic ammonia; however, the litharge and ammonia must then be allowed to work at least twenty-four hours on each other, with very diligent stirring, and, moreover, the litharge must be rubbed very fine.

The ammoniacal solution obtained from the assay is now well stirred, so that it may mix with perfect uniformity with the last washings; then, either the whole, or a part of it, is placed in a clean assay glass, and compared with the standard fluids in similarly formed glass vessels standing on a sheet of white paper. Should it correspond with none of them in the intensity of its colour, the whole of the fluid is diluted somewhat with water, until this is the case. Its volume is thereupon measured in the glass vessel graduated to ounces, &c., and noted. For a check, the dilution may be carried still farther till the colour of the assay corresponds to the next more faintly coloured standard fluid, and then the increased volume be measured anew. This might perhaps be still again repeated, but it becomes more and more uncertain. The calculation of the percentage of copper from the intensity and the volume found, then presents no further difficulty.

Suppose that the assay fluid agrees with the normal solution of four loth of copper to the ounce of water, and its quantity amounts to five ounces, then the quantity of copper in the centner of the assay substance is $5 \times 4 = 20$ loth. This fluid further diluted till it equals the normal solution with three loth of copper, must measure six and two-third ounces if the obtained value of twenty loth is to be confirmed.

According to Heine's experiments, the possible error of observation in the comparison and measurement described, amounts as a maximum with the stronger normal solutions (with sixteen loth and over) to three quarters to one loth, with the weaker ones to scarcely half a loth of copper. In a centner of the assay substance, one loth of copper ·03 per cent. can still be determined with certainty.

Le Play determined in finely pulverised and carefully washed copper slags, the copper in one gramme of the poorest slags to within half a milligramme, and of the richest slags to within one milligramme, by using twenty-six standard fluids with various percentages of copper in cylindrical vessels. The comparison of colours in round vessels is more uncertain than in oblong ones, since in the former the light is dissipated and shadows are produced.

If a substance contains so little copper that the fluid does not equal the most faintly coloured standard fluid in intensity of colour, the assayer must endeavour to remedy the matter by *evaporating* till this is the case. An evaporation is, however, avoided, if possible, first because of the loss of time, and also because other precipitations, carbonate of lime, &c., are apt to be caused by it, and because, when it has to be continued too long, so much ammonia is very apt to be volatilised, that a new addition of it becomes necessary.

This method of assaying soon finds the limits of its accuracy in an increasing percentage of copper in the assay sample, since with fluids rich in copper and therefore strongly coloured blue, the errors of observation soon amount to several loth. And to seek then to better oneself by diluting largely, yields no more accurate results, since a small error of observation in determining the intensity of the colour, is so much the more multiplied in the calculation of the value by the greater number of the ounces.

If *nickel* is contained in the assay substance, the assay cannot be conducted in the way prescribed, since the nickel is extracted by the acids, and dissolves also in caustic ammonia with a blue colour. The assay may also become uncertain from the presence of much *manganese*, *cobalt*, or *chromium*, since they render the hue of the blue colour dingy. *Chromium* may be completely removed by a slight boiling of the ammoniacal fluid; not so cobalt. The presence of *vanadium* or *molybdenum* does no harm.

If *nickel*, or much *cobalt* and *manganese* are contained in the assay substance, the solution obtained by acids and filtered, though not further diluted, must first be decomposed by metallic iron. What is thrown down by the iron is collected on a small filter, washed thoroughly, and then, together with the filter, treated with dilute nitric acid. When the copper is all dissolved, this solution is supersaturated with caustic ammonia and then managed as above. With higher percentages of copper the process of the Swedish copper assay is used for determining the value. The precipitation of the copper may also be performed with hydrosulphuric acid gas.

Le Play removes the injurious influence of manganese, nickel, and cobalt, by allowing the green or violet-coloured ammoniacal solution to stand open to the air for several weeks in a moderately warmed drying furnace, whereby a few variously coloured gelatinous flocks are gradually deposited, and the fluid, after the addition of a few drops of ammonia, then becomes pure blue.

According to Jacquelain and Von Hubert, nickel and cobalt are in a simple way rendered perfectly harmless by gradually adding white pulverised marble to the solution of the assay substance, until the effervescence ceases, and then warming the whole on the sand-bath, whereby all the copper is perfectly precipitated as carbonate, while nickel and cobalt remain dissolved. It is now filtered, washed, the residue dissolved in nitric acid, and the solution treated, as already explained, with ammonia. By the addition of carbonate of potassa to the ammoniacal fluid, and heating, all the manganese precipitates, while the copper remains dissolved in the excess of ammonia, and can be separated from the manganese precipitate by filtration. The manganese must have been present as oxide in the original solution in order that the precipitation by carbonate of potassa may be perfect.

The assayer may convince himself whether *nickel* or *cobalt* is present, by slightly supersaturating a blue ammoniacal solution, obtained by the ordinary process of assaying, with hydrochloric or sulphuric acid, then precipitating the copper *completely* with iron, filtering the residual solution, concentrating somewhat, if necessary, and now supersaturating with ammonia. If the fluid then remains colourless, neither of the two metals is present; a blue colour indicates nickel, a red one cobalt.

Sometimes the normal solutions which when freshly prepared appear azure blue, assume a greenish hue, which renders the comparison difficult, if not impossible. Nitrate of copper produces with ammonia a pure azure blue, sulphate of copper a lilac colour, and chloride of copper greenish hues. Sulphuric and hydrochloric acid are therefore avoided as much as possible in the solution. But, nevertheless, an assay fluid may sometimes, *e.g.* by standing some time in the air, or by slow filtration, become green, in which case the colour is destroyed by a few drops of nitric acid, and ammonia added anew. But sometimes also the greenish colour disappears, if the solution stands in a covered vessel in the air, or by the addition of a few drops of red ammonio-oxide of cobalt.

According to Müller, also, the colour stands in the closest connection with the quantity of ammonia employed, and it therefore leads to greater accuracy in the assay, if a titrated solution of ammonia is used, and the volume of ammoniacal fluid noted, which, after neutralisation of the residual free acid, is used for the solution of the copper. The solution appears more intense when viewed with a grey background than with a white one. A greenish blue colouring becomes the more noticeable, the greater is the excess of ammonia, or the more ammoniacal salts are in the solution.

### 2. JACQUELAIN'S AND VON HUBERT'S COLORIMETRIC ASSAYS.

Heine's method, for the reasons stated, is suitable only for the determination of small quantities of copper. Jacquelain has extended it to the examination of all cupriferous substances, and this process has been further perfected by Von Hubert. According to the latter, a solution of any cupriferous accurately weighed substance is prepared, mixed with ammonia in excess, the ammoniacal solution (assay solution) measured at a definite volume, and a small, likewise measured portion of the measured solution, diluted with water, until its blue colour shows an equal intensity with the blue colour of another solution (normal solution), also cupriferous and ammoniacal, whose copper contents are known once for all. Then, from the quantity of water added, in order to make the two fluids equal to each other in the intensity of their blue colours, the amount of copper in the substance under examination can be determined by calculation.

The normal solution is prepared by dissolving ·5 of a gramme of chemically pure copper in dilute nitric acid, adding ammonia in excess, and diluting with distilled water until the whole at 12° C. amounts to one litre=1000 cubic centimetres. The solution is filtered, and preserved in a flask provided with a glass stopper ground in to fit it.

For the preparation of the assay fluid, with substances whose percentage of copper ranges from 1·5 to the highest per cent., two grammes, and with the poorer substances five grammes, are brought into ammoniacal solution with the precautions specified in Heine's assay. This solution, with over five per cent. of copper, is measured at two hundred cubic centimetres, with two to five per cent. of copper at one hundred and fifty cubic centimetres, and with two per cent. and under, at one hundred; and also, as may be required, at 90, 80, 60, 50 c.c., according to the intensity of the fluid. Only with an extremely small quantity of copper is the assay fluid evaporated to a smaller volume, in order to be able to conduct the colorimetric test with accuracy.

The comparison of the intensity of colour of the assay fluid with the normal fluid is accomplished in two different ways, according as the former, when measured at a definite volume, is darker or lighter than the latter. This can be seen if a small arbitrary portion of each is poured into a glass tube of nine millimetres interior diameter, twelve centimetres in length, and uniform thickness, and the two tubes are held in parallel positions over a piece of white paper so that they rest firmly on it, and are inclined to it at an angle of about 45°, and direct light falls upon them. Shadow should not fall upon the tubes.

*a. The Assay Fluid is Darker than the Normal Solution.*—By means of a pipette, five cubic centimetres of the normal solution are placed in a glass tube closed at the bottom and not graduated, and seven millimetres in interior diameter and twelve centimetres long. Since 1000 c. c. of the normal solution contain ·5 of a gramme of copper, five cubic centimetres contain exactly ·0025 and the ratio 5 : ·0025 expresses once for all the known proportion of copper in the normal solution.

Five cubic centimetres of the definitely measured assay fluid are now also placed in a beaker and gradually diluted with water till they show the same intensity of colour as the normal solution. In the comparison the assay fluid must be in a similar tube to that containing the normal solution. With richer proportions a greater accuracy is attained in this comparison, if the assay fluid is so far diluted that its intensity still appears as little as possible darker than that of the normal solution, and then water added carefully, and drop by drop, till its intensity is judged as little as possible lighter than that of the normal solution, whereupon the mean of the two volumes noted is taken as the correct value. The measuring of the diluted assay fluid is performed in glass tubes of nine millimetres interior diameter and fifty centimetres in length, which from their lower closed end to the circular mark designated by 0, hold exactly five cubic centimetres, and from 0 upwards are divided into cubic centimetres and their tenths.

If, for example, two grammes of the assay substance have been weighed out, the assay fluid measured at 200 cubic centimetres, and five cubic centimetres of it diluted to 8·2 cubic centimetres, in order to obtain an equal intensity of colour between the normal and assay fluid, then the percentage of copper, *x*, follows from this according to the following chain of ratios :—

| | |
|---|---|
| *x* | 100 per cent. |
| 2 | 200 c. c. assay fluid. |
| 5 | 8·2 c. c. diluted assay = normal solution. |
| 5 | ·0025 grammes of copper in normal solution. |
| *x* | = 8·2 per cent. of copper. |

*b. The Assay Fluid is Lighter than the Normal Solution.*— In this case five cubic centimetres of the normal solution are diluted till their intensity is equal to that of the assay solution that has been measured at a definite volume, and for the comparison larger tubes of nine millimetres interior diameter are used.

If, for example, two grammes of the assay substance have been weighed out, 150 cubic centimetres of assay fluid obtained from it, and to get the same intensity of colour, five cubic centimetres of the normal solution diluted to 8·4 cubic centimetres, the quantity of copper *x* amounts, according to the following chain of ratios, to 2·205 per cent. :—

| | |
|---|---|
| *x* | 100 per cent. |
| 2 | 150 c. c. assay solution. |
| 8·4 | 5 c. c. normal solution. |
| 5 | ·0025 grammes of copper. |
| *x* = 16·8 | 37·5 = 2·205 per cent. |

This assay is adapted for all cupriferous substances, since nickel, cobalt, and manganese, which would influence the result unfavourably, can be removed without particular difficulty. It is also easy to be learned by those less practised in analytical operations, can be completed in a few hours, and is far less expensive than the dry assay. From two to one tenth per cent. of copper can also be determined by it with accuracy.

Heine, however, prefers his method when a small percentage of copper is to be determined, since by it even one loth of copper in the centner = ·03 per cent. can be determined, and there is less liability to error. While in slag assays with nine to eighteen loth of copper in the centner, by Heine's method, errors of half a loth are not to be avoided, variations of more than one loth occur by Von Hubert's process. The latter works with a too deeply coloured normal fluid, corresponding to a solution of over fourteen loth of copper to one ounce of water, while Heine does not exceed four loth. The process is surer if the fluids are diluted and thicker layers of them compared, and thus the hue made artificially deeper, than if small quantities of stronger fluids are compared and the hue made artificially lighter by comparing them in thinner layers, or especially in tubes, where the light is dispersed and shade produced. The comparison in oblong glasses is therefore to be preferred to that in tubes.

By a comparison of Von Hubert's assay with that of the Oberhartz, it appears that, as Von Hubert's experiments themselves have shown, both give equally accurate results for substances not too poor in copper (*i.e.* containing not less than 5 per cent.). The Oberhartz assay allows a direct determination of the copper, requires less apparatus, is also very simple, and can be completed in a shorter time. Since different individuals are differently susceptible to colours, and the blue colour of the ammonio-oxide of copper, in consequence of causes yet unknown, sometimes inclines more or less to greenish, and thereby renders observation difficult, therefore, for the sake of greater certainty, though not of greater accuracy, those assays by which a determination of the copper is possible by weight, should in general be preferred to the colorimetric methods, and this is the case with the Oberhartz assay down to two per cent. With smaller percentages the colorimetric assay must be called to our aid. It is not yet settled that with higher percentages of copper the principle of colorimetry is a correct one; that is, that the intensity of the colour is directly proportional to the quantity of the colouring agent.

Since ammoniacal solutions poor in copper often show a dash of greenish, Von Hubert prepares a normal solution for such by dissolving one decigramme of copper and diluting to one litre of fluid.

### 3. A. Müller's Assay with the Complementary Colorimeter.

Müller has sought to remove the uncertainties in the comparison of colours which arise in direct inspection, by a new colorimeter, which is based upon the neutralisation of the colour to be measured, by its complementary colour. For cupriferous ammoniacal fluids a more or less reddish yellow is suitable, according to their character.

This process, for the detailed management of which reference must be made to the publications of Müller, consists essentially in determining in millimetres the height of the column of fluid of a dilute ammoniacal solution with a known percentage of copper, which, when the laterally falling light is cut off with a complementarily coloured glass plate placed over the fluid, will produce the neutralisation point, *i.e.* white light. For every testing of an assay fluid instituted with the same glass plate, the percentage of colouring matter then follows in inverse proportion to the actual height of the column of fluid. If the volume of the assay fluid is also measured, which is directly done with great accuracy in the apparatus itself, all the data are obtained for the calculation of the amount of copper in the assay substance, after the necessary corrections, constant for each instrument, have been taken into consideration.

As the experiments of Heine, and also investigations made in the metallurgical laboratory at Clausthal, have demonstrated, large and small quantities of copper can be quickly determined with great accuracy by means of this colorimeter.

## *d.* VOLUMETRIC COPPER ASSAYS.

### 1. PELOUZE'S COPPER ASSAY [PRECIPITATION ANALYSIS] WITH SULPHIDE OF SODIUM.

*Pelouzé's Process.*—This is dependent on the decolorisation of an ammoniacal solution of copper by sulphide of sodium. The standard solution of sulphide of sodium may be made by dissolving four ounces of crystallised sulphide of sodium in a quart of water. To determine the strength of this solution proceed as follows:—

Dissolve 20 grains of pure copper in nitric acid, dilute the solution with water, add excess of ammonia, and make up the deep blue solution thus afforded to about half a pint, which introduce into a suitable flask, and heat to ebullition. Whilst the contents of the flask are in process of boiling, pour into a burette, divided into 100 parts, 100 measures of the solution of sulphide of sodium, and when the cupreous solution is boiling, gradually add the sulphide of sodium until the liquid in the flask becomes colourless: it must be kept in a constant state of ebullition. When this is the case, the whole of the copper is thrown down as a black precipitate of oxysulphide of copper ($5CuS,CuO$).

The number of degrees of solution of sulphide of sodium required to produce this effect must be noted, and the numbers so used are equivalent to and represent 20 grains of copper.

Suppose 186 measures or degrees had been necessary, then as

$$186 : 1 :: 20 : x = \frac{20 \times 1}{186} = 0{\cdot}1075$$

so that every division or degree in the burette corresponds to 0·1075 grain of copper; and in the assay proper the operator has only to multiply the number of divisions used by the number obtained as above, and the result will be the amount of copper in the quantity of ore or other material operated on.

The assay of the ore is thus made:—50 grains are dissolved in nitric acid, or in nitro-hydrochloric acid (aqua regia), as may be found most advantageous. When the solution is complete, the flask is allowed to cool, water added, and then considerable excess of ammonia. If the precipitate thus produced be very bulky, it must be separated by filtration, well washed, and the washings added to the filtrate; if not, the small amount need not be separated. The solution must now be boiled, and the sulphide of sodium added as just described. When the blue colour of the solution in the flask has disappeared, the number of divisions is noted, and multiplied by the number obtained in standardising the solution as already described. The result is the quantity of copper in 50 grains: this multiplied by 2 gives the percentage.

Pelouze made a great number of experiments to ascertain how far the presence of other metals might interfere with the accuracy of this process, and his results assure him that nickel and cobalt alone have any injurious effects; and fortunately these occur but seldom, and in small quantities, in copper ores and their products.

### 2. C. Kunsel's Volumetric Method for the Estimation of Copper and Nickel and Copper and Zinc.

Dr. C. Kunsel has proposed the following plan of estimating copper volumetrically.

With careful manipulation, the author states that the error, at the most, will be, in the case of copper, $\frac{1}{4}$ per cent. in the case of nickel, $\frac{3}{4}$ per cent.; and in the case of zinc $\frac{1}{2}$ per cent.

An ammoniacal solution containing $\frac{1}{10000}$th of copper reacts distinctly on moist, recently precipitated sulphide of zinc, the zinc dissolving while the copper is precipitated in the form of sulphide. The sulphides of zinc and nickel decompose instantly in a hot ammoniacal solution of copper. A solution which contains $\frac{1}{20000}$th of sulphide of sodium reacts distinctly on an ammoniacal silver solution or a solution of nitro-prusside of sodium.

Starting from these three reactions, the author proposes the following volumetrical methods for nickel and copper or copper and zinc:—

1. *Sulphide of Sodium Solution.*—It being necessary to make use of pure sulphide of sodium, the author prepares it by saturating a solution of caustic soda free from carbonate, with sulphuretted hydrogen, and driving off the excess of the gas. The solution is then diluted so that a cubic centimetre precipitates a centigramme of copper or nickel.

2. *Standard Test: Preparation of Standard Solution of Sulphide of Sodium for Estimation of Copper.*—The author dissolves a known weight of pure copper in nitric acid, supersaturates the solution with ammonia, dilutes it, and heats it to boiling. The solution of sulphide of sodium is then added to the hot solution of copper, stirring continually

until a drop of the mixed solution no longer colours sulphide of zinc brown. Sulphide of zinc for indicating the complete precipitation of the copper is prepared in the following way:—Zinc is dissolved in hydrochloric acid, the solution is supersaturated with ammonia, and is then boiled with a little sulphide of zinc to remove the lead which is always present in commercial zinc. The ammoniacal solution of zinc, now free from lead, is filtered, and decomposed with sulphide of sodium, a small quantity of zinc being allowed to remain in solution. The moist sulphide of zinc, with excess of zinc solution, is then spread evenly upon filter paper several layers thick. When the paper has absorbed most of the solution, the moist white layer of sulphide of zinc is ready for use.

3. *Test Solution for Nickel.*—The strength of the solution for nickel is ascertained in a similar way to the preceding. A known weight of nickel is dissolved in acid, the solution is treated with excess of ammonia, diluted with water, and the solution of sulphide added until a drop of the mixed solutions colours an ammoniacal solution of silver pale brown, or gives a red with nitro-prusside of sodium; *i. e.* until all the nickel is precipitated, and a slight excess of the sulphide is present in the mixture. But as freshly precipitated sulphide of nickel will colour the silver solution brown and redden the nitro-prusside, it is necessary to separate the precipitate before the solution is tested. The author therefore filters a drop by placing it on a strip of filtering paper, and applies the silver solution or nitro-prusside to the moist spot on the opposite side of the paper.

4. *Test Solution for Zinc.*—In the case of zinc the author recommends pure chloride of nickel to be used to indicate the complete precipitation. The strength of the solution of sulphide of sodium having been ascertained in the experiment with copper, it may be obtained for nickel and zinc by calculation.

5. *Process for Copper and Nickel.*—The ore or alloy freed from arsenic is dissolved in hydrochloric acid, with the addition of some nitric acid, and, when necessary, is evaporated to dryness, the deposit dissolved in hot water, and

filtered to separate silica. Any iron may be removed from the mixed chlorides by the addition of ammonia. The solution, freed from iron, is then rendered strongly ammoniacal, heated to boiling, and the standard solution of sulphide of sodium added (with continual shaking), until a drop of the mixed solutions no longer acts on sulphide of zinc, *i.e.* until all the copper is precipitated. The number of cubic centimetres of the standard solution is then read off, and the amount of copper calculated. The addition of the standard solution is now continued until there is a trace of the sulphide in excess in the mixture, which is ascertained in the manner before described; the additional number of cubic centimetres employed will give the amount of nickel.

6. *Process for Copper and Zinc.*—This may be the same as the foregoing process for copper and nickel, making use of chloride of nickel to indicate the complete precipitation of the zinc.

### 3. PARKES' AND MOHR'S METHOD BY CYANIDE OF POTASSIUM.

In carrying out the volumetric estimation according to the directions of Mr. Parkes, a solution of cyanide of potassium is slowly added to a blue ammoniacal solution of copper, when the latter gradually loses its colour, and finally becomes quite colourless; upon this chemical reaction the estimation of copper by cyanide of potassium depends. By ascertaining by direct experiment the amount of cyanide of potassium solution required to discharge the colour in an ammoniacal solution containing a given weight of copper, it is easy by a comparative experiment to determine the amount of copper in a given weight of ore.

For the preparation of the standard solution 2,000 grains of fused cyanide of potassium are to be dissolved in two quarts of water, to produce a solution of which 1,000 grains measure will be equal to about ten grains of metallic copper. The solution should be preserved in green glass stoppered bottles, and kept as much as possible away from the light; it is liable to a slow decomposition, which will necessitate the standard being checked at intervals of one or two weeks. In order to standardise the solution, a burette, holding 1,000 grain measures, is filled to the zero mark, and a piece of pure electrotype copper previously cleaned by means of dilute nitric acid, washed and dried, is accurately weighed. About eight grains may be conveniently taken; this is dissolved in a pint flask by dilute nitric acid, and, after the energy of the first action has subsided, the solution is warmed and ultimately boiled to expel all the nitrous acid fumes. It is diluted with cold water to the bulk of nearly half-a-pint, treated with ammonia in excess, and to the deep blue solution the cyanide is added from the burette until the colour is so nearly discharged that a faint lilac tint only remains. This will generally become quite bleached on standing at rest for a short time, so that the cyanide must not be added too hastily towards the end of the operation. It will be advisable to control the standard by a second experiment upon another weighed portion of copper, and to stop short of bleaching entirely the faint lilac tint of the solution. A piece of white paper folded and placed under and behind the flask during the decolorisation, will aid in recognising the proper tint of the solution.

In applying this process to the examination of copper ores, a known weight of the finely-powdered sample is introduced into a beaker provided with a glass cover, and moistened with strong sulphuric acid; strong nitric acid is then added, and the whole digested on a sand-bath until nitrous fumes are no longer given off. Should a small quantity of sulphur be separated in the treatment of pyrites ores, the small globules may be taken out, burnt, and the residual copper dissolved in a few drops of nitric acid and mixed with the remainder. Water is now to be added and left in contact for a short time to extract all the metallic salt from the insoluble residue, which need not be filtered off; and so, likewise, when ammonia is added in the next place, any peroxide of iron which may thus be precipitated is left in the solution, for it is apt to contain a small proportion of copper when first thrown down; but this is entirely removed by the cyanide of potassium later in the experiment.

When the ore contains much iron it is considered desirable to remove the hydrated peroxide by filtration, in order to be enabled to determine with greater precision the last effects of the cyanide of potassium; and in the event of requiring to know the amount of iron present in the ore, the precipitated peroxide on the filter is re-dissolved in dilute sulphuric acid, reduced to the state of protoxide by metallic zinc, and then tested in the usual way with a standard solution of permanganate of potash.

The metals which interfere with this mode of valuing copper ores, are silver, nickel, cobalt, and zinc. The first may readily be separated by adding a few drops of hydrochloric acid to the original solution; the other metals may be excluded by following one of the methods pointed out by the author for that purpose.

### 4. SCHWARZ'S METHOD WITH THE MODIFICATION OF F. MOHR.

·634 grammes of the assay substance are dissolved in water or acid, the solution placed in a flask, the excess of acid neutralised with carbonate of soda, a small quantity of neutral tartrate of potash and caustic potash or soda added, till all is dissolved to a deep blue fluid. If this does not take place at once, more tartrate of potash is added. The solution is warmed to 40—50° R., and pure starch- or honey-sugar, or pure white honey, added, with frequent shaking, whereby the oxide of copper is gradually reduced, and finally a fire-red precipitate of suboxide of copper produced, so that the fluid assumes a yellowish colour. With too strong boiling the precipitate becomes brownish-red. The diluted fluid is filtered, the precipitate well washed with hot water, and together with the filter put into a wide-necked flask, and a suitable quantity of chloride of sodium, and then hydrochloric acid, added, whereupon the suboxide of copper dissolves to colourless subchloride of copper, forming an easily soluble double salt with the chloride of sodium. Without removing the filter, titrated solution of permanganate of potash is then added to the solution from a Gay-Lussac burette, with constant stirring, until its rose-colour, which is destroyed by the oxidation of the suboxide of copper, again appears. The permanganate of potash solution is so titrated that 100 cubic centimetres of it correspond to ·56 gramme of iron. The cubic centimetres used then give the copper in per cents.

The calculation depends upon the fact that one atom of suboxide of copper, $Cu_2O$, takes one atom of oxygen from the permanganate of potash to form oxide of copper, and two atoms of protoxide of iron, which contain two atoms of iron, also take one atom of oxygen to form sesquioxide of iron; hence one atom of iron corresponds to one atom of copper, or twenty-eight of iron to 31·7 of copper, so that from the percentage of iron which the titrated solution of permanganate of potash gives, the percentage of copper may be calculated.

### 5. E. O. Brown's Method by Hyposulphite of Soda.

The process described by Mr. E. O. Brown is particularly applicable to the determination of copper in gun-metal, brass, and other alloys which contain no large amounts of iron and lead. It is founded on the reactions between salts of copper and the neutral iodides, and on the conversion of the liberated iodine into hydriodic and tetrathionic acids by a standard solution of hyposulphite of soda.

These reactions may be thus expressed:—

$$2(CuO,\bar{A}) + 2KI = Cu_2I + I + 2(KO,\bar{A})$$
$$I + 2(NaO,S_2O_2) = NaI + NaO,S_4O_5$$

The completion of the second reaction is manifested by the bleaching effect produced upon the blue iodide of starch by the addition of the hyposulphite. A convenient strength of solution for this purpose may be made by dissolving 1,300 grains of the crystallised salt in two quarts of water. The iodide of potassium must be free from iodate; and a clear solution of starch employed.

From 8 to 10 grains of the copper or alloy are dissolved in dilute nitric acid, and the red nitrous fumes expelled by boiling. The nitrate of copper is converted into acetate by adding carbonate of soda until a portion of copper remains precipitated, and then re-dissolving in acetic acid. The solution is diluted with water, and about 60 grains of iodide of potassium in the form of crystals dropped into the flask, and allowed to dissolve. The standard solution of hyposulphite of soda is now poured in from a burette, until the greater part of the dark-coloured free iodine disappears. A little of the starch solution is now added to make its presence more apparent, and the addition of the hyposulphite continued until the bleaching is completed, when the pale yellow colour of the subiodide of copper will alone be visible. The amount of copper in the ore or alloy is calculated from the number of divisions indicated upon the burette.

Copper ores containing much iron (which interferes by reason of the dark red colour of the acetate) may be dissolved in nitric acid, and treated with sulphuretted hydrogen to precipitate the copper, the sulphide being collected on a filter, washed, and re-dissolved in nitric acid to produce a solution suitable for testing by this process. Or the hyposulphite may itself be employed to furnish a precipitate of disulphide of copper.

### 6. Fleck's Modification of Mohr's Method.*

The proposal to take the action of solution of cyanide of potassium on ammoniacal solution of copper, as the foundation of a method for estimating copper, is due to Carl Mohr.†

The azure blue colour disappears, $Cu_2Cy,NH_4Cy$ and KO are formed, while 1 eq. of cyanogen is separated, which acting on the free ammonia, gives urea, oxalate of urea, cyanide of ammonium, and formiate of ammonia (Liebig‡).

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* This process is given by Fresenius, condensed from Polytechn. Centralbl. 1859, 1313.

† Annal. d. Chem. u. Pharm. 94, 198; Fr. Mohr's Lehrbuch der Titriermethode, 2, 91.

‡ Annal. d. Chem. u. Pharm. 95, 118.

The decomposition is not always the same: the quantity and degree of concentration of the ammonia has a marked influence on it, from which it appears that neutral ammonia salts also affect the results.

Fleck proposes the following modification:—

Instead of caustic ammonia, use a solution of sesquicarbonate of ammonia (1 in 10), warm the mixture to about 60°, and in order to render the end reaction plainer, add 2 drops of solution of ferrocyanide of potassium (1 in 20); the blue colour of the solution is not altered by this addition, nor is its clearness affected. The value of the cyanide of potassium solution is first determined, by means of copper solution of known strength, and it is then employed on the copper solution to be examined. On dropping the cyanide of potassium into the blue solution warmed to 60°, the odour of cyanogen is plainly perceptible, and the colour gradually disappears. As soon as the ammoniacal double salt of copper is destroyed, the solution becomes red from the formation of ferrocyanide of copper, without any precipitate appearing, and with the addition of a final drop of cyanide of potassium this red colour in its turn vanishes, so that the fluid now appears quite colourless.

The method thus modified yields, it is true, better, but still only approximate results.* Where such are good enough, the method is certainly convenient.

### 7. Fleitmann's Method.

Mr. Sutton gives the following description:—

The metallic solution, free from nitric acid, bismuth, or lead, is precipitated with zinc; the copper collected, washed, and dissolved in a mixture of perchloride of iron and hydrochloric acid; a little carbonate of soda may be added to expel the atmospheric air. The reaction is—

$$Cu + Fe_2Cl_3 = CuCl + 2FeCl.$$

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* In six experiments, in which he had purposely added different quantities of carbonate of ammonia, Fleck used for 100 c. c. copper solution, in the minimum 15·2, in the maximum 15·75, in the mean 15·46 c. c. cyanide of potassium solution.

1 eq. of copper, therefore, produces 2 eq. protochloride of iron. When the copper is all dissolved, the solution is diluted and titrated with permanganate; 56 iron represent 31·7 copper.

If the original solution contains nitric acid, bismuth, or lead, the decomposition by zinc must take place in ammoniacal solution, from which the precipitates of either of the above metals have been removed by filtration; the zinc must be finely divided and the mixture warmed; the copper is all precipitated when the colour of the solution has disappeared. It is washed first with hot water, then with weak hydrochloric or sulphuric acid and water, to remove the zinc; again with water, and then dissolved in the acid and perchloride of iron as before.

### *e.* OTHER COPPER ASSAYS, BY LEVOL, BYER AND ROBERT, RIVOT, AND WOLCOTT GIBBS.

1. Levol treats a solution of ammonio-oxide of copper, with exclusion of air, with a weighed strip of copper, by which the oxide of copper is reduced to suboxide. From the loss which the strip of copper suffers, the quantity of copper in the solution can be calculated. This assay is indeed accurate, if no other substances are present which oxidise copper, but it requires several days' time, and the copper is determined by a difference, which is always more uncertain than a direct determination.

A similar assay, in which, however, instead of an ammoniacal solution, a hydrochloric acid solution is used, has been given by Runge.

2. Robert and Byer precipitate the copper from its solution, by a simple galvanic apparatus, on a weighed plate of copper, whose increase in weight then gives the quantity of copper in the assay. The operation lasts ten to twelve hours, and no other similarly precipitable metals should be present.

3. Rivot precipitates the *suboxide* of copper from its solution as sulphocyanide of copper $Cu_2Cy_2S$, and from the weight of this salt calculates the amount of copper directly,

or after it has been converted into $Cu_2S$ by igniting with sulphur in a covered porcelain crucible at a red heat.

This method is indeed accurate, and generally practicable, but it requires a complete acquaintance with the analytical operations and the observance of a mass of small precautions, so that it does not differ from an analytical process.

Professor Chapman, of Toronto, gives the following directions for the detection of minute traces of copper in iron pyrites and other bodies:—

Although an exceedingly small percentage of copper may be detected in blowpipe experiments by the reducing process as well as by the azure blue coloration of the flame when the test-matter is moistened with chlorhydric acid, these methods fail in certain extreme cases to give satisfactory results. It often happens that veins of iron pyrites lead at greater depths to copper pyrites. In this case, according to the experience of the writer, the iron pyrites will almost invariably hold minute traces of copper. Hence the desirability, on exploring expeditions more especially, of some ready test by which, without the necessity of employing acids or other bulky and difficultly portable reagents, these traces of copper may be detected.* The following simple method will be found to answer the purpose:—The test substance, in powder, must first be roasted on charcoal, or, better, on a fragment of porcelain,† in order to drive off the sulphur. A small portion of the roasted ore is then to be fused on platinum wire with phosphor-salt; and some bisulphate of potash is to be added to the glass (without this being removed from the wire) in two or three successive portions, or until the glass becomes more or less saturated. This effected, the bead is to be shaken off the platinum loop into a small capsule, and treated with boiling water, by which either the whole or the greater part will be dissolved; and the solution is finally to be tested with a small fragment of ferrocyanide of potassium ('yellow prussiate'). If copper be present in more than traces, this reagent, it is well known, will produce a deep red precipitate. If the copper be present in smaller quantity—that is, in exceedingly minute traces—the precipitate will be brown or brownish-black; and if copper be entirely absent, the precipitate will be blue or green—assuming, of course, that iron pyrites or some other ferruginous substance is operated upon. In this experiment the preliminary fusion with phosphor-salt greatly facilitates the after solution of the substance in bisulphate of potash. In some instances, indeed, no solution takes place if this preliminary treatment with phosphor-salt be omitted.

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* In blowpipe practice—as far, at least, as this is possible—the operator should make it an essential aim to render himself independent of the use of mineral acids and other liquid and inconvenient reagents of a similar character. If these reagents cannot be dispensed with altogether, their use, by improved processes, may be greatly limited.

† In the roasting of metallic sulphides, &c., the writer has employed, for some years, small fragments of Berlin or Meissen porcelain, such as result from the breakage of crucibles and other vessels of that material. The test substance is crushed to powder, moistened slightly, and spread over the surface of the porcelain; and when the operation is finished, the powder is easily scraped off by the point of a knife-blade or small steel spatula. In roasting operations, rarely more than a dull red heat is required; but these porcelain fragments may be rendered white-hot, if such be necessary, without risk of fracture.—*Canadian Journal*, September 1860.

Dr. Wolcott Gibbs recommends the electrolytic precipitation of copper and nickel as a method of analysis. He says:—

The precipitation of copper by zinc, in a platinum vessel, with the precautions recommended by Fresenius, leaves nothing to be desired, so far as accuracy, ease, and rapidity of execution are concerned. The method labours, however, under a single disadvantage—the introduction of zinc renders it difficult, or at least inconvenient, to determine with accuracy other elements which may be present with the copper. It has occurred to me that this difficulty might be overcome, the principle of the method being still retained, by precipitating the copper by electrolysis with a separate rheomotor. The following numerical results, which are due to Mr. E. V. M'Candless, will satisfactorily show the advantages of the method for the particular cases in which it is desirable to employ it. The copper was in each case in the form of sulphate: the deposition took place in a small platinum capsule, which was made to form the negative electrode of a Bunsen's battery of one or two cells, in rather feeble action. The positive electrode consisted of a stout platinum wire, plunged into the surface of the solution of copper at its centre. The following table gives the results obtained in the analysis of pure sulphate of copper:—

| Number | Salt taken | Copper found | Percentage |
|---|---|---|---|
| 1 | 1·2375 | 0 3145 | 25·41 |
| 2 | 0·4235 | 0·1075 | 25·38 |
| 3 | 1·0640 | 0·2705 | 25·42 |
| 4 | 1·3580 | 0·3440 | 25·33 |
| 5 | 0·5665 | 0·1450 | 25·59 |
| 6 | 0·4735 | 0·1205 | 25·48 |

In seven determinations of copper in the alloy of copper and nickel employed by the Government for small coins the following results were obtained:—

| Number | Weight of Alloy | Copper | Percentage |
|---|---|---|---|
| 1 | 0·4160 | 0·3640 | 87·50 |
| 2 | 0·6180 | 0·5410 | 87·54 |
| 3 | 0·4600 | 0·4090 | 88·91 |
| 4 | 0·5120 | 0·4481 | 87·51 |
| 5 | 0·4220 | 0·3693 | 87·51 |
| 6 | 0·2525 | 0·2225 | 88·11 |
| 7 | 0·3705 | 0·3255 | 87·85 |

The percentage of copper required by the formula CuO, $SO_3 + 5HO$ is 25·42, while the Government standard alloy of nickel and copper contains 87·50 per cent. of copper. The time required for precipitation varied from one to three hours, the separation of the last traces of copper being in each case determined by testing a drop of the liquid upon a porcelain plate with sulphuretted hydrogen water. The copper after precipitation was washed with distilled water, dried in vacuo over sulphuric acid, and weighed with the platinum vessel. The only precaution necessary is to regulate the strength of the current so that the copper may be precipitated as a compact and bright metallic coating, and to dry as quickly as possible. When the copper is thrown down in a spongy condition, it not only oxidises rapidly, but it is impossible to wash out the last traces of foreign matter contained in the solution. This is well shown by No. 3 and No. 4 of the second series, in both of which cases the copper was precipitated too rapidly. The solution from which the copper has been deposited contains the other elements present in the original substance. It may be easily poured off without loss, and the washings added.

It appears at least probable that nickel may be determined by electrolysis in the same manner as copper, the solution employed being the ammoniacal sulphate with excess of free ammonia. Mr. M'Candless obtained in two determinations in a commercial sample 91·36 and 91·60 per cent. of nickel. In both cases the nickel was thrown down completely as a bright, coherent, metallic coating upon the platinum.

## BLOWPIPE REACTIONS OF COPPER.

### Minerals of Copper.

**Sulphide of Copper.**—*Alone*, on charcoal, gives off sulphurous acid, fusing readily in the outer flame. In the inner flame it is covered with a crust, and does not fuse.

In the *open tube* sulphurous acid is disengaged, but no sublimate is produced. The residue, treated with soda and borax, gives a button of copper.

**Argentiferous Sulphide of Copper.**—*Alone*, fuses easily, giving off sulphurous acid. Cupelled with lead, on bone-ash, it leaves a large bead of silver, and the cupel appears a blackish green.

**Sulphide of Antimony and Copper, Bournonite.**—*Alone*, in the open tube, gives off the antimonial smoke, with an odour of sulphurous acid. A slip of Brazil-wood paper, on being placed within the tube, is bleached.

*On charcoal*, a deposit of antimony, but no trace of lead. The bead diminishes in size, becoming grey, and semi-malleable. Fused with soda, it gives a grain of copper.

**Copper Pyrites, Sulphide of Iron and Copper.**—*Alone*, on being heated, blackens, becomes red by cooling, and fuses more easily than the sulphide of copper, finally giving a bead attractable by the magnet. This bead is brittle, and reddish-grey in the fracture. If after a long exposure to the oxidising flame it be treated with a small quantity of borax, a regulus of copper is obtained.

In the *open tube*, much sulphurous acid is given off.

*With Soda*, reduced iron and globules of copper are obtained, provided the ore has been sufficiently roasted.

**Sulphide of Tin and Copper, Tin Pyrites.**—Beforet he blowpipe it becomes, by roasting, covered with a snow-white powder, which is oxide of tin. The white powder also encircles the globule to the extent of about two lines.

In the *open tube*, sulphurous acid is given off.

**Needle-ore, Aikenite.**—*Alone*, it fuses, giving off vapour, which coats the charcoal snow-white, slightly yellowish on the interior edge, finally giving a metallic bead resembling bismuth.

In the *open tube* it gives off a white smoke, one part of which is fusible, and the other volatile. The first part is converted by fusion into limpid drops, which become white by cooling; there is also an odour of sulphurous acid. Treated by fluxes, the resulting bead of bismuth gives the reaction of copper. After a long blast, a grain of copper may be obtained, which by cupellation with lead gives traces of silver. A fusible white smoke, at the commencement of the operation, indicates the presence of tellurium.

**Chloride of Copper.**—*Alone*, colours the flame blue, with greenish edges. A red pulverulent deposit forms on the charcoal around the assay; the fused matter reduces, giving a grain of copper, surrounded by slag.

With fluxes, the chloride behaves as the oxide.

**Carbonate of Copper.**—*Alone*, in the matrass, gives water, and blackens.

On charcoal it fuses, and behaves like oxide of copper.

**Arseniate of Copper** behaves with fluxes in the same manner as the oxide of copper, but exhales a strong odour of arsenic, and gives, when reduced with soda, a white and brittle bead.

**Oxide of Copper.**—*Alone*, in the oxidising flame, it is fused into a black bead, which is reduced on charcoal. In the reducing flame, at a temperature which does not suffice to fuse copper, the oxide is reduced, and shines with the lustre characteristic of metallic copper; but as soon as the blast ceases the metal re-oxidises, and becomes black or brown.

Exposed to a stronger heat, it gives a bead of metallic copper on fusion.

*With borax*, oxide of copper readily fuses in the oxidising flame, forming a beautiful green glass, which loses its colour in the reducing flame, but which on cooling becomes cinnabar-red and opaque. If the oxide of copper be impure, the glass is generally deep brown, and only becomes opaque in an intermittent flame.

*With microcosmic salt* it fuses, attended with the same phenomena as with borax. If the quantity of copper be small, the glass occasionally becomes transparent and ruby-coloured in the reducing flame; this change takes place at the instant of solidification. Commonly the glass becomes red and opaque, similar in appearance to an enamel.

When the quantity of the copper is so small that the character of the red oxide cannot be made evident in the reducing flame, a small quantity of tin must be added, and the flame kept up only for an instant. The glass, previously colourless, becomes red and opaque by cooling. If the blast be kept up too long, the colour is destroyed, owing to the reduction of the copper.

*With soda*, on the platinum wire, a beautiful green glass is formed, which becomes opaque and colourless on cooling. On charcoal it is absorbed, and the oxide reduced. The blowpipe is capable of detecting a smaller quantity of copper than any other test; especially when it is not in combination with other metals, which by their reduction would disguise its presence. In the latter case we must use borax and tin. When copper and iron are associated together, a single assay separates them into distinct particles; the one may be told by colour, and the other by being attracted by the magnet.

It is difficult, by borax or microcosmic salt, to determine this copper in slags as protoxide or suboxide, on account of the small quantity generally present; and, moreover, the other ingredients, which are chiefly silicates of different earths and difficultly reducible metallic oxides, destroy the reaction of oxide of copper. For this reason, instead of employing the *reduction* process, the slags must be treated with soda on charcoal. If by this method, also, copper should not be detected, a greater quantity, about 100 milligrammes, must be reduced with its own quantity of soda, half of borax, and 30 to 50 milligrammes proof lead, and the lead, united to a globule, treated with boracic acid till all is dissolved, or the copper, is concentrated. If the slag contains a trace of copper, this becomes reduced, and combines with the lead, and, in the first case, colours the boracic acid red, green, or blue. If the copper present is very minute, the tinge is seen on those parts only where the latter part of the lead containing copper was dissolved.

A small quantity of copper contained in a substance can often be detected, if not in combination with sulphuric acid, by one or two drops of hydrochloric acid. It is only necessary to moisten the substance with this acid, and heat it in the forceps, in the azure of the blue flame, when, by this means, the outer flame is coloured greenish-blue, and often reddish-blue, by the chloride of copper formed.

# CHAPTER X.*

## ASSAY OF LEAD.

All minerals and substances containing lead may, for the purposes of the asssayer by the dry way, be divided into four classes :—

*Class I.* comprises sulphides, antimonial or otherwise (galena, &c.).

*Class II.* includes all plumbiferous substances containing neither sulphur nor arsenic, or mere traces only of these elements (litharge, minium, carbonate of lead, native and artificial, lead fume, cupel bottoms, furnace hearths, lead slag, &c.).

*Class III.* comprises all substances into whose composition either sulphuric, arsenic, chromic, or phosphoric acid, or a mixture of either, enters (pyromorphite, wolframite, &c.).

*Class IV.* Alloys of lead.

### CLASS I.

Before describing the different modes of assaying substances of this class, it will be as well to pass in review the action of various reagents on sulphides of lead, in order that the *rationale* of the assay of those ores may be better appreciated.

*Action of Oxygen.*—If galena be roasted at a very gentle temperature, care being taken to avoid fusion, it will be converted into a mixture of oxide of lead and sulphate of lead, with evolution of sulphurous acid, thus :—

$$2(PbS)+7O=PbO+PbO,SO_3+SO_2.$$

*Action of Metallic Iron.*—This metal completely and readily decomposes sulphide of lead, giving metallic lead in a pure state, thus :—

$$PbS+Fe=Pb+FeS.$$

On the one side we have sulphide of lead and metallic iron, on the other metallic lead and sulphide of iron.

*The Alkalies and Alkaline Carbonates* decompose sulphide of lead, but only partially; pure lead is separated, and at the same time a very fusible grey slag is formed, which contains an alkaline sulphate and a compound of sulphide of lead and an alkaline sulphide. A certain proportion of the alkali is reduced by the sulphur, which is converted into sulphuric acid, so that no oxide of lead is produced. This reaction may be thus expressed :—

$$7(PbS)+4(KO)=4Pb+KO,SO_3+3(PbS,KS).$$

*Nitrate of Potash* completely decomposes sulphide of lead, with the reduction of metallic lead and formation of sulphate of potash and sulphurous acid, thus :—

$$2(PbS)+KO,NO_5=2Pb+KO,SO_3+SO_2+N.$$

If the nitre be in excess, the lead will be oxidised in proportion to the excess present, and if there be a sufficiency added, no metallic lead at all will be produced.

*Argol.*—The presence of carbonaceous matter much favours the decomposition of galena, by determining the reduction of a larger quantity of potassium to the metallic state, and thereby the formation of a larger quantity of alkaline sulphide. With 4 parts of argol to 1 part of sulphide, 80 parts of lead are reduced. If the reaction were complete, the decomposition would be as follows :—

$$PbS+KO+C=Pb+KS+CO.$$

For the reactions of oxide of lead (litharge) and the sulphate of lead on sulphide of lead, see pages 179 and 181.

From the reactions above given, it will be seen that there are many substances capable of completely reducing the lead from its sulphide, and yet few can be used safely with any advantage, as so to use them would imply a knowledge of how much sulphur and lead were in the ore to be assayed, in order to tell the precise quantity of either of the reagents required; for it is evident that if either more or less of some were added, a faulty result would be the consequence: so that some systematic mode of assay, which may be suitable for all classes of galena, whether mixed with other sulphides or with gangue, must be contrived. To facilitate this we now proceed to give an outline of the processes generally adopted in the assay of lead ores by various persons.

### 1. FUSION WITH CARBONATE OF POTASH.

This plan is used at the Oberhartz, and described by Kerl as follows:—

One centner of the very finely pulverised assay substance is weighed out, mixed with three to four times its weight of pure, dry, and finely pulverised carbonate of potash, and covered over, in a small clay crucible (fig. 58) with a layer of decrepitated chloride of sodium about one-fourth of an inch thick. The assays thus prepared are placed in the thoroughly heated muffle of a large assay furnace (figs. 21, 22) having a strong draught. They remain in the highest temperature of the furnace, with the mouth of the muffle closed with glowing coals, till they have come into perfect fusion (about twenty to thirty minutes). The draught opening is then closed, and at the same time the muffle opened, until the temperature has fallen so far that the crucibles appear brownish-red, and the vapours above them have greatly diminished, or have disappeared. At this heat the crucibles, whose contents must, however, always remain in perfect fusion, are maintained, according to the fusibility and composition of the assay sample, and the draught of the furnace for a longer or shorter time (ten to twenty-five, generally ten minutes). This period during which the heat is allowed to remain low, is called the *cooling* of the assay.

The furnace is now again brought back to its first temperature, by completely opening the draught and closing the muffle. Ten to fifteen minutes of this last heating are in most cases sufficient. Only poor ores, &c., which contain also a pretty large quantity of arsenic, or of the sulphides of iron, zinc, and copper, are allowed to continue hot five to ten minutes longer.

If one has many assays to make, it will be found advantageous to mix those which contain larger quantities of foreign sulphides, or, by reason of their earthy contents, are difficultly fusible, with more or less *borax*, or, instead of this, to place them in the back and hotter part of the muffle, while those that are very rich in lead and easily fusible, are placed in front, since the latter will be hot enough here, and more easily reached by the air than those deeper in the muffle.

The crucibles, when cold, are broken, the lead buttons obtained are freed from all adhering slag or substance of the crucible, and if the assay were otherwise successful, their weight determined. The assays should not be too rapidly cooled, because the slag is thus easily cracked, and the still half-fluid button lying below is apt to be broken into several pieces.

In a successful assay, the lead melted together to a button, deports itself under the hammer and knife like pure lead, and possesses also its colour. If the slag shows, upon its surface of separation from the metallic button, lead grey spots with metallic lustre, it will generally also be found that a thin layer of not completely decomposed glistening sulphide or subsulphide of lead has at the same time deposited itself upon the button. This layer, if the above appearance presents itself in a high degree, can be rubbed off or removed in fine scales. The lead button itself then shows upon its surface a high metallic lustre, which does not have the colour of pure lead, but a darker and blackish hue. Assays of this kind are to be rejected; they have not been allowed to remain *cool* long enough, or they have in the process become *too* cold; they give the amount of lead too low, and often very considerably so. In assays, which have stood too long in the furnace in the last fusing heat, a very bright button of lead is also found; but here the layer of undecomposed sulphide of lead is wanting, as also the glistening spots on the surface of the slag surrounding the button. If the influence of the heat and air continues too long, then besides a loss through volatilisation of the lead, a slagging of the oxide of lead may take place. A button that is brittle, laminated, and brilliantly white in the fracture, indicates an insufficiency of flux, or the presence of antimony and arsenic. In successful assays the lead button generally has a bluish appearance which, although not dull, is at the same time not strongly brilliant. The slag must be completely homogeneous, and must have settled down uniformly towards the bottom of the crucible, so that it does not stick in a thick layer to the upper part of the sides of the crucible. It shows by this that it has been in proper fusion. It must have covered over the button in a thick layer (about one-fourth of an inch thick). The chloride of sodium covering, or a more or less colourless slag that is formed, containing chloride of sodium and carbonate of potash, overlies in a still thicker layer the true dark-coloured slag containing the foreign metallic oxides. A porous slag containing metallic globules indicates a small quantity of flux or too low a temperature; a brilliant vitreous slag, too high a temperature and a slagging of lead. An assay and its duplicate, must, moreover, give equal results.

*Lead matt* and *lead fume* are smelted, with the addition of borax and coal-dust, with carbonate of potassa, and with the first the heat is allowed to last somewhat longer (perhaps to three-quarters of an hour) than with ores. The carbonate of potassa assay gives for lead matt, with its not inconsiderable lead contents (thirty per cent. and over) pretty satisfactory results.

The theory of this lead assay appears from the following.

If perfectly pure galena is intimately mixed with three or four times its weight of good dry carbonate of potash, placed in a clay retort, and this so arranged in the muffle of the assay furnace that its neck projects from the mouth of the muffle, while in the opening of the neck a glass tube is closely fitted, which goes into a receiver, from which it is further prolonged in a second tube, it will be observed that at first only a little water collects in the receiver, proceeding from the small quantity of moisture always present in the carbonate of potash. Later, with an incipient red heat in the retort, a gas is disengaged, which upon closer investigation proves to be pure carbonic acid gas, i.e. free from sulphurous acid (means of proof:—absence of odour; conducting of the gas through a solution of manganate of potash, reddened by sulphuric acid; through lime and baryta water, and through a solution of caustic potash, and further examination of the solution of salts obtained). The disengagement of gas becomes more active with a stronger red heat, without yielding gases of different composition, but ceases again after a while. In order to obtain assurance of a complete decomposition, the retort may be kept for an hour at a very strong red heat. After the cooling and breaking of the retort, some pure oxide and carbonate of lead is found deposited in the neck of it, then a pure lead button upon the bottom, and over this a brown slag, free from little globules of lead. It consists in by far the greatest part of sulphide of potassium and still undecomposed carbonate of potash, but also in small part of silicate of potash derived from the silica of the retort. If this slag is treated with water till nothing further will dissolve, the substances named can be easily shown to exist in the solution. The solution is colourless, and when supersaturated with acids disengages sulphuretted hydrogen, but throws down no sulphur. In the treatment of the slag with water, sulphide of lead remains behind in black flocks, even the exterior character of which shows that it is not undecomposed galena, but sulphide of lead separated from a chemical combination.

If the brown slag from the retort is placed in a small uncovered crucible and brought back into the hot muffle of the assay furnace and melted, then after some time, whether the slag was covered with chloride of sodium or not, a button of lead again separates at the bottom of the crucible, and the brown slag now shows itself decolorised. If the crucible is removed from the furnace too soon, only the upper layer of slag is decolorised, and that lying below is still completely unchanged. The decolorised slag consists of carbonate and sulphate of potash, and no longer contains any trace of sulphide of potassium.

In the above described lead assay, the process in the strong preliminary heat proceeds as in the retort, i.e. the potash of the carbonate of potash is reduced to potassium, while it yields its oxygen to the sulphur of the galena and with it forms sulphuric acid; the liberated potassium takes up sulphur from another portion of galena, forming sulphide of potassium. The galena would now in this double way soon lose all its sulphur, if a combination—a sulphur salt—of sulphide of potassium with sulphide of lead did not form, which resists all further action of the carbonate of potash [$4\,(KO,CO_2) + 7PbS = 4Pb + 3\,(KS,PbS) + KO,SO_3 + 4\,CO_2$]. The carbonic acid of the thus decomposed carbonate of potash escapes together with that set free by the sulphuric acid formed, and causes a puffing up of the mass, by which globules of lead already separated are raised up with it, and may perhaps remain with some of the slag sticking to the upper crucible walls. They would here oxidise and produce yellow spots. The covering of chloride of sodium is designed to guard against loss of lead in this and similiar ways. It serves in a certain manner to rinse down the sides of the crucible.

The atmospheric oxygen, in the open crucible, is not entirely excluded by the covering of chloride of sodium. In the *cooling* of the assay, it oxidises the sulphur salt contained in the upper part of the slag, forming sulphate of potash and a portion of sulphate of lead. The latter, during the last high heat, decomposes the sulphide of lead still remaining in the slag, in such a way as to produce metallic lead. ($PbS + PbO,SO_3 = 2Pb\ \ 2SO_2$.) The reduced particles of lead separate well from the slag thus rendered thinly fluid. Matts must be allowed to *cool* longer than ores.

The carbonate of potash assay presupposes in general great practice, and close attention on the part of the assayer; and moreover, if one wishes to find the correct value at once, without fruitless preliminary examinations, and without the necessity of repeating the assay, a general knowledge of the constituents of the assay sample, so far, for example, as this can be obtained by the aid of mineralogy, is necessary.

The assay after this method, which requires but little preparation, can only be conducted in the muffle furnace, but then in pretty large number (as many as fifty at once). For its success it is indispensably necessary that the *cooling* of the assay be undertaken and stopped again at the right time and in the proper degree. If it is allowed to *cool* too long, too much sulphate of lead is formed in proportion to the sulphide of lead still present in the slag, and in the last heating up, by the action of the two upon each other, easily scorifiable oxide of lead is produced. ($PbS + 3PbO, SO_3 = 4PbO + 4SO_2$.) If the *cooling* is too soon interrupted, only a small part of the sulphide of lead in the sulphur salt is oxidised, and by the action of the oxidised portion upon the sulphide of lead subsulphide of lead is produced, which either remains in the slag or settles upon the lead button. ($2 PbS + PbO,SO_3 = Pb_2S + 2Pb + 2SO_2$.) Experience gives the only means at hand to guide us here, but leaves us easily in the lurch, so that the result of the assay becomes more doubtful than in some of the methods hereafter described.

With substances containing *antimony* this assay deserves the preference over the others, since most of the antimony remains in the slag in the state of sulphide and oxide. An addition of saltpetre works advantageously. *Arsenic* and *sulphide of arsenic* mostly go off in fumes during the smelting, but nevertheless always cause the formation of a brittle metallic button. *Sulphide of copper* remains in great part in the slag, but a part of the copper is desulphurised and goes into the lead. If the quantity of copper present is very considerable, the button of metal may be considered as black copper, and refined, and the loss thereby occurring, reckoned as lead.

*Proto-sulphide of iron*, which occurs, for example, in lead matt, is decomposed by carbonate of potash, forming metallic iron, which desulphurises the galena. *Iron pyrites*, on the other hand, occasions the forming of a large quantity of sulphide of potassium, and in consequence of this, of a sulphur salt.

It follows, therefore, from the above, that ores, which contain much foreign sulphides, are not suited to this method of assaying, since they cause the production of a large amount of sulphide of potassium, which always retains sulphide of lead. By an addition of saltpetre to the carbonate of potash, these sulphides may, indeed, be partially decomposed: only an oxidation of the lead is apt to be produced, as well as a mechanical loss by the violent action of the saltpetre.

Bredberg has, in his comparative investigations of the different methods of assaying lead, pronounced the smelting of the raw ore with carbonate of potash and chloride of sodium, to be the most inapplicable of all. He cannot, however, have understood the theory of this method, since he melted his assays in the crucible furnace, and, therefore, *without* access of air, and that in his investigations he must, therefore, have found the quantity of lead much too small, is perfectly evident from the above. Thus his opinion in relation to this method must have proved erroneous, and this may be mentioned here for the reason that many a verdict against this method of assaying has originated in the same or a similar mistake.

(56 p.c. carbonate

From pure galena, by the carbonate of potash assay, eighty per cent. of lead at most can be obtained. Calcined *carbonate of soda* is inferior to carbonate of potash as a desulphurising agent, and always yields a few per cent. less lead than the latter. According to Phillips, seventy-five to seventy-seven per cent. of lead are obtained from galena with carbonate of soda. With *cyanide of potassium*, under certain circumstances, the same result can be obtained as with carbonate of potash, and it does not require so high nor so long continued a temperature; still it offers no real advantage over carbonate of potash. An addition of thirty to thirty-five per cent. of saltpetre to an assay, with which ten parts of carbonate of soda are used, promotes, indeed, the desulphurising of the lead, but also increases the loss of lead.

At the Oberhartz smelting-house, the lead button is weighed out to pounds, and a difference of five pounds is allowed between different assayers. It is also a custom, though not a correct one, to allow as many pounds difference as there are tens of pounds in the weight of the lead button obtained. Thus, with a lead contents of thirty and seventy pounds, the difference in the separate assays might amount to three and seven pounds respectively.

### 2. FUSION WITH BLACK FLUX.

A modification of the preceding method of assaying, which is sometimes employed, consists in using, instead of the carbonate of potash, an equal quantity of black flux, or indeed of argol, or in mixing a few per cent. of powdered charcoal or flour with the carbonate of potash, or in replacing it in part by argol. Too great an addition of carbon diminishes the fusibility of the mass, and hinders the flowing together of the separated particles of lead. By using argol the operation lasts longer, because the mass remains pasty until most of the tartaric acid has been decomposed : but a greater product of lead is obtained. The chemical reaction during the operation is thereby modified so that the carbon of the black flux exerts an influence upon the potassa, and partially reduces it to potassium ; the potassium, thus set free, works now as before upon the galena. The latter is thus without the influence of the air more completely decomposed than by carbonate of potash alone, and the smelting is, therefore, conducted in covered crucibles (fig. 58) in the wind furnace. But since there also sulphide of potassium is formed, and this dissolves sulphide of lead, it is more advisable, for the completest possible separation of the lead, to perform the smelting in open crucibles in the muffle, in order to allow the atmospheric oxygen to work at the same time on the assay. The product of lead from pure galena does not generally exceed seventy-six to seventy-nine per cent.

At the Victor-Frederick smelting works in the Hartz, one centner = one hundred and fourteen pounds of galena, is mixed with three or four times as much black flux, and with pyritic ores ten pounds of borax-glass are added. The mixture is covered with chloride of sodium, heated for about twenty-five minutes in the muffle furnace with a charcoal fire, and then, after the mouth of the muffle has been opened for about five minutes, taken out of the furnace.

## 3. FUSION WITH METALLIC IRON.

Schlutter and many of the older assayers were aware that iron would desulphurise galena, and ever after advised the addition of a certain quantity of that metal to the different fluxes, which they used in lead assays; but it was at the practical School of Mines, at Montiers, that iron was first employed alone.

The process is extremely convenient and easy of execution; it always succeeds, and requires no troublesome attention. The fusion takes place quietly, without frothing or bubbling: and as the whole substance employed requires but little space, very small pots may be employed, or a very large quantity assayed. But this process can only be employed for pure galenas, or those which contain at most a few per cent. of gangue.

When galena is heated with iron, the metal is transformed into protosulphide, from whence it follows, that to desulphurise galena 22·6 per cent. is required; but experience has shown that it is better to employ a little more, and 30 per cent. can be used without inconvenience. The iron employed ought to be in the state of filings, or wire cut very small. The mixture is placed in a crucible, which is three-fourths filled: the whole is covered with a layer of salt, carbonate of soda, or black flux, and exposed to a full red heat. After the flux is perfectly fused, the pot may be cooled and broken, and a button is obtained, which at first sight has a homogeneous aspect, but on being struck with the hammer separates into two distinct parts. The lower part is ductile lead: the upper a very brittle matt, of a deep bronze colour, and slightly magnetic. Pure galena yields, by this process, 72 to 79 per cent. of lead, so that there is a considerable loss, which loss is entirely due to volatilisation. Berthier says that it does not appear possible to avoid this loss, which amounts from 6 to 13 per cent., giving as a reason that it is probable galena begins to sublime before it arrives at the proper heat for decomposition.

Antimonial galenas, or galenas mixed with iron pyrites, may be assayed in the same manner; but then a sufficiency of iron must be added to reduce the antimony to the metallic state, as well as to reduce the iron pyrites to the minimum of sulphuration. If the galena be mixed with blende, the greater portion remains in the slag, because it is only decomposed by iron at a very high temperature. Blende being infusible by itself, much diminishes the fusibility of the matts produced; and if it exists in very large quantity, it can even hinder their full fusion; in which case, some protosulphide of iron and metallic iron must be added to the assay, to make the slag more fusible.

All minerals are at a minimum of sulphuration, when existing in matts from metallurgical works; therefore, much less iron may be used in their assay than if they were pure ores. In very rich lead matts, in which the lead exists as subsulphide, from 10 to 12 per cent. is sufficient. A small excess of iron may be employed without inconvenience; but if a larger proportion be added than is necessary to execute the desulphuration, the matt contains some iron in the metallic state, and loses its liquidity, and in consequence retains some globules of lead.

The usual mode of assaying lead ores (galena) in the lead mills is by a modification of this process: in lieu of placing the ore in an earthern crucible, and adding nails or filings, a given weight of the ore is projected into a red-hot wrought-iron crucible, which is kept in the fire for about a quarter of an hour, or until all the galena seems decomposed. The lead thus reduced is poured into a mould; and if the scoriaceous matter be not well fused, the iron crucible is returned to the fire and heated still more strongly, and any lead that may be separated is poured into the mould and weighed with the rest. This is a very rude and imperfect process, and gives only tolerable results with pure galenas, but is perfectly unsatisfactory with those containing much earthy matter, as not above half the lead is obtained, owing to volatilisation and exposure to the air, and the loss of globules in the slag. This process succeeds much better when a flux is added: this may be argol, or carbonate of soda, or a mixture of both (see next process).

### 4. FUSION WITH CARBONATE OF SODA, OR BLACK FLUX AND METALLIC IRON.

When galena is heated with an alkaline flux, out of contact of air, the slag contains a double sulphide of lead and the alkaline metal employed: if iron be thrown into this fused mixture, metallic lead separates, and the iron combines with the sulphur formerly combined with the lead, and the slag will contain a double alkaline sulphide, containing sulphide of iron instead of sulphide of lead, thus:

$$PbS + KS + Fe = Pb + FeS + KS.$$

Any earthy substances the ore may contain will be dissolved by the alkaline flux, without very much impairing its fluidity. All these facts being considered, it may be readily seen that the assay of all earthy bodies containing sulphide of lead may be made in this manner, with as much accuracy as this method of assay can be capable of. Either carbonate of soda or black flux may be employed as the alkaline reagent, and more of either of those substances must be employed, in proportion to the increased quantity of earthy matters the ore contains. Two parts are nearly always more than sufficient for poor ores, and are best for all cases, because an excess of flux does not diminish the yield of lead; nevertheless, it is sometimes convenient to employ, for the latter class, but half a part. As to the iron, it is employed only to separate that part of the lead which has been dissolved in the state of sulphide by the alkali, but not decomposed; so that much less may be employed than is necessary for the decomposition of the whole amount.

Experiment has shown that the maximum amount of lead from pure galena may be obtained by the use of the following mixtures:—

2 parts of black flux, or carbonate of soda, and 10 to 12 per cent. of iron.

1 part of black flux, or carbonate of soda, and 20 per cent. of iron.

½ a part of black flux, or carbonate of soda, and from 25 to 30 per cent. of iron.

When black flux is employed, and the iron is in the state of filings, it would be inconvenient to employ too much of the latter, especially if the assay were heated very strongly, because the button of lead might be contaminated with iron; but when carbonate of soda is used with small iron nails instead of filings, the excess of iron is not inconvenient, but rather useful, because the desulphuration is certain to be complete.*

The following changes take place in both cases. That portion of iron filings mixed with the carbonate of soda which has not been sulphuretted, is reduced to the state of oxide by the carbonic acid of the alkaline carbonate, and remains combined or suspended in the slag; so that the proportion of iron is never too great, and never becomes mixed with the lead. When black flux is employed, the same oxidation does not take place, on account of the presence of carbonaceous matter, so that the portion of filings not combined with sulphur, and which is merely held in suspension in the flux, passes through it with the globules of lead to the bottom of the crucible; but if, instead of filings, small nails are employed, they only suffer corrosion at their surface, without change of form or softening, and after the assay are found fixed in the surface of the button of lead, so that they can be detached very readily, and without loss of lead.† This, however, I have found no easy task, and have always sustained a notable loss.

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* The French assayers use a piece of plate iron in the shape of a horse-shoe, which is moved about in the melted mass until no more globules of lead attach themselves to it.

In Germany a piece of iron-wire is used. What iron is not consumed by the assay is found still hanging together in a single mass.

† Berthier.

## 5. ROASTING AND REDUCING ASSAY.

This mode is preferable for ores and substances which contain a considerable quantity of foreign sulphides, or arsenides and antimonides, and a greater or less amount of earthy matter. It is used in many parts of Germany (at the Unterhartz), and described by Kerl thus:—

Two assay centner of ore, matt, &c., are heated at first at a low red heat in the muffle, on a roasting dish that has been previously rubbed with chalk. After ten to fifteen minutes, they are taken out of the furnace, then again roasted at a moderate temperature for ten or fifteen minutes with frequent turning of the dish. The assay is then once more taken from the furnace, allowed to cool, rubbed up in an agate mortar, and again roasted for half an hour, whereupon it is taken out of the furnace; tallow is added while it yet glows, and it is again brought to a strong red heat. The rubbing up and calcining with tallow is repeated several times more, and when afterwards the assays shall have been exposed for two hours continuously to a strong red heat, with the mouth of the muffle almost entirely closed, if no more sulphurous acid vapours escape, the roasting is considered as finished. This lasts from six to twelve hours. The roasted sample is then portioned out with the balance, each portion mixed with three or four parts of black flux and an equal quantity of borax and glass, placed in a small crucible covered with chloride of sodium, furnished with a little piece of coal as a cover, and smelted in the wind furnace, for about a quarter of an hour after the fire is well ignited. Assays that have worked well, give nearly equal malleable buttons that do not contain matt, and a black uniformly fused slag.

The purpose of the roasting is to convert the metallic sulphides, arsenides, and antimonides into oxides. But since in the process, sulphates, antimoniates, and arseniates are produced, we seek to destroy these by repeated calcining with tallow (see above), instead of an intermixture of coal-dust or flour. By melting the roasted assay with its charge at *not too high* a temperature, the oxide of lead is reduced, and the foreign oxides and earths contained in the sample are, by the aid of the potash in the black flux, as well as of the borax and glass, slagged off. If sulphates or sulphides have remained behind in the roasted ore, they will in the smelting be partially desulphurised by the action of the oxides, especially of the oxide of iron. An addition of metallic iron would in this respect be advantageous.

The roasting is a lengthy process, and one which causes not an unimportant loss of lead. If it is not done thoroughly, then, in the reduction smelting, sulphur salts are formed, which always retain lead, as also a plumbiferous matt, which surrounds the lead button. By the use of too high a temperature in the smelting, a great part of the foreign oxides is reduced, and the lead becomes contaminated. This reduction, however, cannot be entirely avoided, even with a rightly conducted temperature.

Galena melts indeed less easily if the air is excluded, than metallic lead; but is much more volatile than the latter, and is decomposed by fusion into a higher sulphide which is volatile and a lower one ($Pb_2S$) which remains as a residue. By roasting, galena gives a mixture of oxide and sulphate of lead, from which last the sulphuric acid cannot be separated, even at a fusing temperature. Sulphate of lead becomes soft by heat, fuses at a bright white heat, and is converted by carbon, with a considerable loss of lead through volatilisation, into oxide of lead, metallic lead, or subsulphide of lead, according to the quantity of carbon used, and the temperature employed. With oxide of lead, the sulphate easily fuses together.

### 6. ASSAY WITH SULPHURIC ACID.

The assay sample is rubbed as fine as possible. A suitable quantity of it is then weighed out for an assay, and boiled with four to eight times its weight of oil of vitriol until all is decomposed. All excess of sulphuric acid is then evaporated in a porcelain capsule, under a flue with a good draught, and the mass carried to dryness. Boiling sulphuric acid decomposes the sulphides, changing iron, copper, nickel, zinc, &c., into salts which dissolve readily in water, and also at the same time changing the sulphide of lead into sulphate, which in water, especially in water that is cold, and also contains free sulphuric acid, is practically insoluble. The composition of the ore is in general ascertained by first heating it with nitric acid or aqua regia, and then, with the addition of sulphuric acid, evaporating to dryness. The dry mass, when cold, is moistened with a small quantity of sulphuric aid, then cold water; it is afterwards, by the aid of a small brush, brought without loss upon a small filter, and washed with cold water until the filtrate is colourless. Unnecessary prolonging of the washing is to be avoided, for sulphate of lead is not absolutely insoluble. The filter, with its contents, is dried in the funnel, until it can be easily taken out of it without tearing. It is now laid immediately into the clay crucible, in which the sulphate of lead is afterwards to be reduced, and this is placed in a very gentle stove warmth. (Some carbonate of potash may be first poured into the bottom of the crucible.) When completely dry, the crucible with the cover laid over it is very gently heated, so that the filter carbonises, which very soon happens, as the free sulphuric acid is not completely soaked out. The filter is now stirred up with a little rod, black flux or carbonate of potash with coal-dust and iron are introduced into the crucible, and intimately mixed with the sulphate of lead and the rest of the insoluble residue. About four or five times the volume of the whole residue are taken of black flux, and the assay is further treated, as prescribed in the portion which follows upon the assaying of sulphate of lead.

In this way the lead is concentrated, and the foreign sulphides, which were specified above as the cause of the failure of the assay in such cases, completely removed. The result obtained in this way is satisfactory, and deserves the same confidence as one obtained in favourable circumstances by the ordinary lead assay from an ore with a medium or high percentage of lead.

## CLASS II.

*Assay of Substances of the Second Class.*—The assay of these substances is very simple indeed. Litharge, minium, carbonate of lead, &c., may be assayed by simple fusion with carbonaceous matter: but when the operation is thus conducted loss of lead is sustained: it is therefore better to add some flux which will readily fuse, and allow the globules of reduced lead to collect into one button. No flux fulfils this condition better than a mixture of carbonate of soda and argol, which is to be intimately mixed with the assay. The following is the best mode of procedure:—To 200 grains of the finely-pulverised substance add 100 grains of argol, and 300 of carbonate of soda, and intimately mix; place the mixture in a crucible which it about half fills, and cover with a layer of common salt about $\frac{1}{4}$ inch thick; submit the crucible to a very gradually increasing temperature, keeping the heat at low redness for about a quarter of an hour; then urging it to bright red until the contents of the crucible flow freely; take it from the fire and shake, tap it as directed in the copper assay, and either pour the contents into the mould or allow to cool in the crucible. If the operator be pressed for time, the mould may be used, but it is recommended to allow the assay to cool in the crucible, for unless the operator be very careful, and have had some considerable practice, he is very liable to lose a small quantity of metal in the pouring. After the contents of the mould or crucible, as the case may be, are cold, the lead may he separated from the slag by repeated gentle blows from the hammer: if any of the slag or crucible adhere to the button, the latter may be readily freed from it by placing the button between the finger and thumb with its edge on the anvil, and then gently hammering it. The lead will be so altered in shape under the hammer that the slag or crucible readily falls off; and by continuing the process, the whole may be removed. The cleaned button may then be hammered into a cubical form, and is ready for weighing.

In the assay of lead great care must be taken in the management of the temperature, as lead is sensibly volatile above a bright red heat, even when covered with flux, and still more so if any portion be uncovered from want of sufficient quantity of flux; neither must the assay remain in after the flux flows freely, for a loss may thereby occur from oxidation, by decomposition of carbonate of soda, as explained in the reduction of copper ores and the copper-refining process.

For the *rationale* of this mode of assay, refer to page 192, which explains the decomposition of oxide of lead, with the production of metallic lead, carbonic acid, and water, by the agency of a substance, like argol, containing both carbon and hydrogen.

Cupel bottoms, some lead fumes, and siliceous slags, require a modified treatment in their assay, as the substances mixed with the oxide of lead (more particularly bone-ash in the cupel bottoms) are very infusible; and if the flux already mentioned as applicable to the other matters belonging to this class were employed, a very high temperature would be necessary; and as lead, as already stated, is sensibly volatile above a bright red heat, an evident loss of that metal would be the result.

Cupel bottoms may be thus assayed: 400 grains of the finely-pulverised bottoms to be mixed with 200 grains of argol, 400 grains of carbonate of soda, and 400 grains of pulverised fused borax; the mixture placed in a crucible as already directed, covered with salt, and the fusion conducted as just described.

Lead fumes and siliceous slags require only half their weight of fused borax, with 200 argol, 400 carbonate of soda, and 400 substance (fume or slag) covered with salt.

The addition of the borax, which is a most powerful flux, causes the fusion of the assay to take place almost as readily with the last-named intractable substances, as with the former easily fusible and reducible matters. The assay, however, is rather more subject to ebullition or boiling over the sides of the crucible; hence it must be carefully watched, and the instant it appears likely to do so the crucible must be removed from the fire, gently tapped on the furnace top, and when the effervescence has subsided returned to the furnace, and this operation repeated until the fusion proceeds tranquilly.

The lead obtained in these assays, if the ore or substance contained any foreign metal, is never pure: if silver, copper, tin, or antimony be present, the whole of either of these metals will be found alloyed with the lead produced; but if the ore contains zinc, and it be heated sufficiently, but a trace remains; nevertheless the zinc carries off with it a considerable quantity of lead.

The following experiments will show what an influence the presence of zinc has upon the return of lead:—

100 parts of litharge,
100 parts of oxide of zinc,
300 parts of black flux,

were fused together, and 84 parts of lead were the result.

100 parts of litharge,
100 parts of oxide of zinc,
600 parts of black flux,

were fused together, and but 70 parts of lead were produced, instead of 90, which the pure litharge ought to have given. Hence it will be seen that, the more zinc is reduced, the more lead is volatilised.

If oxide of iron be present in the assay, it is reduced, but it remains in suspension in the slag, and the lead does not contain a trace when it has not been too strongly heated. If the assay be made at a very high temperature, the iron may be fused, and then the lead will be ferruginous; this may be ascertained by means of the magnet. A similar result was obtained by many assayers, who thought for a long time that lead and iron could thus combine together; but by careful examination it is easily ascertained that the ferruginous buttons are but mechanical mixtures of lead and iron in grains. Indeed, by careful hammering, nearly all the iron may be removed from the lead, so that it loses its magnetic properties.

The oxides of maganese, when mixed with the ore, are changed into protoxide, which remains in the flux, and is not reduced.

*Humid Assay of Ores of the Second Class.*—Pulverise the substance very finely, and to 100 grains placed in a flask add one ounce of nitric acid diluted with two ounces of water (if minium be the substance to be analysed, it must be first heated to redness, so as to reduce the whole of the lead it contains to the state of protoxide), and gently heat, gradually raising the temperature to the boiling-point: when all action seems to have ceased, pour the contents of the flask into an evaporating basin, and evaporate to dryness with the precautions directed in the analysis of iron ore Allow the dry mass to cool, and a little dilute nitric acid, gently warm for an hour, then add water, boil, and filter. The whole of the lead now exists in the solution as nitrate: thus, say carbonate of lead had been the substance under analysis, then—

$$PbO,CO_2+NO_5=PbO,NO_5+CO_2$$

To the filtered solution containing the nitrate as above, add solution of sulphate of soda, or dilute sulphuric acid, until no further precipitation takes place: insoluble sulphate of lead will now be thrown down: this must be allowed to completely subside by standing in a warm place; and when the supernatant liquid is quite bright the sulphate may be collected on a filter, washed, dried in the water-bath, and weighed. It contains 68·28 per cent. of metallic lead.

The decomposition of the nitrate of lead by sulphate of soda may be thus expressed—

$$Pb+O,NO\ +NaO,SO_3=PbO,SO_3NaO,NCO_5.$$

Determination of lead by standard solution will be described at the end of this chapter.

## CLASS III.

*Assay of Substances of the Third Class.*—In the assay of bodies belonging to this class, a reducing agent must be employed: but if that alone be used, the sulphates and arseniates produce sulphides and arsenides, and not pure lead. The action of another reagent is therefore necessary, in order to deprive the lead of sulphur and arsenic with which it is combined.

There are two reagents known for the sulphates—they are the alkaline carbonates or metallic iron; but for the arseniates and arsenites iron must be employed, because the alkaline carbonates have no action on the arsenides.

In all cases black flux is employed: this furnishes a reducing agent for the oxides, and a flux for the earthy matters. Iron is added when the arsenites or arseniates are assayed; but that metal may either be employed or not, when the sulphates are operated upon. It is, however, always better to use it.

When a mixture of black flux and iron is employed, the assay is made in exactly the same manner as that of the sulphides (large nails are preferable whenever the use of iron is indicated in a lead assay). With the sulphate, the sulphide of iron formed combines in the slag with the alkaline sulphide; but it is not so with the arseniates and arsenites. The arsenide produced mixes neither with the lead nor the slag, but gives rise to the formation of a brittle matter which adheres slightly to the button of lead.

When only black flux is employed, either of the two following processes may be adopted:—First, the ore can be fused with four parts of common black flux; then, as in the case of sulphide, the excess of carbon determines the formation of a large quantity of an alkaline sulphide; and consequently produces a desulphuration of the lead. Secondly, it may be fused with such a proportion of black flux, containing only the requisite proportion of carbon to reduce the oxide of lead, or with an equivalent mixture of carbonate of soda and charcoal. Pure sulphate of lead fused with one part of carbonate of soda and four per cent. of charcoal gives 66 of lead; but in order to employ this method the richness of the ore must be known, and the dry way is then useless, excepting for the estimation of the silver these substances always contain.

*Humid Assay of Substances of the Third Class.*—These are treated in precisely the same manner as those of the preceding class.

## CLASS IV.

## ALLOYS OF LEAD.

### ASSAY WITH SULPHURIC ACID.

No docimastic assay is known for exhibiting the lead isolated from its alloys. In individual cases a serviceable result may be attained, if the metal (*e.g.* of copper by refining *it*) with which the lead is combined be determined, and its quantity then deducted. This method is, however, in general, the more unreliable, the smaller is the quantity of lead, or when the lead is alloyed with several metals; so that then the quantity of lead can often only be determined by the partial or complete aid of the wet way.

For many products (*e.g.* crude lead, hard lead—containing antimony or arsenic—plumbiferous copper, &c.), the assay with sulphuric acid described on page 388 is suitable. One assay centner of the substance is decomposed by nitric acid or aqua regia, then, with the addition of sulphuric acid, evaporated to dryness, and the dry mass treated as above directed. If the residue consists only of sulphate of lead, it can be brought upon a weighed filter, and from the weight of the residue after drying, the amount of lead may be calculated. 100 parts sulphate of lead contain 68·33 parts lead.

### ADDITIONAL REMARKS ON THE LEAD ASSAY.

#### *Comparison of the Different Methods for the Docimastic Determination of Lead in their application to various Products.*

Markus has made the following comparative experiments with the methods of assaying lead ores most in use at the Austrian smelting works at Joachimsthal.

*a. Assay with Black Flux and Iron.*—One assay centner (5·7 grammes) of the finely rubbed, sifted, and dried assay substance was mixed with two assay centuer of black flux made of sixteen saltpetre and forty argol, and sixty pounds of borax-glass in a mixing capsule, and put into a clay crucible, on the bottom of which a piece of thick iron wire 1″ inch long and forty centner in weight, had been placed in a vertical position. The crucible charge, covered over with two centner of decrepitated chloride of sodium, was smelted in a mineral coal muffle furnace, with the mouth of the muffle closed, and the draught half open, at a moderate temperature, the temperature then lowered for six to seven minutes by opening the mouth of the muffle, then the muffle closed again for an equal period, and the final heat then given. The cessation of the low crackling of the assay was now carefully attended to, and this, ceasing after seven to eight minutes, indicated the completion of the assay. The duration of the assay, by the way, was twenty minutes.

*b. Roasting and Reduction Assay with Iron.*—One assay centner of galena was roasted, at first with a low temperature, for about thirty minutes on a roasting dish, and the dish then pushed into the back part of the muffle for six to eight minutes to destroy the sulphates formed. The roasted ore was rubbed fine, intimately mixed with three hundred centner of black flux and fifty centner of borax-glass, placed in a crucible with a piece of iron weighing twenty centner at the bottom, covered with salt, and smelted as above.

*c. Roasting and Fusing with Black Flux.*—One centner of the roasted ore was smelted as before with three hundred centner of black flux and fifty centner of borax, but without iron.

The results obtained proved—

1. That with all those products which contain tolerably pure sulphide of lead, especially with high percentages, the iron assay, *a*, gives in a remarkably predominant degree the most lead (as high as ninety-six per cent. of all the lead present).

2. With impure lead ores, which contain more foreign sulphides, the assay *a* gives likewise the highest percentage, though the assays *b* and *c* give only a few per cent. less.

3. If foreign sulphides are present in predominant quantity, the methods of *b* and *c* give a slightly higher percentage than that of *a*.

### *Levol's Fusion Assay with Ferrocyanide and Cyanide of Potassium.*

According to Levol, the method of assaying galena for its lead by smelting it with black flux and iron is defective in two respects. First, it is difficult to choose precisely the quantity of iron required for the reduction of the lead, and a lack or excess of it either gives too little lead or a button containing iron; and second, in order that the reaction may be complete and the lead unite to a button, we are compelled to use a very high temperature, at which lead volatilises. The first defect can indeed be removed by the use of iron crucibles, but these are easily rendered unserviceable, and require a pouring out of the fused mass, and then globules of lead are apt to remain in the slag.

By the use of a mixture of fifty parts of cyanide of potassium and one hundred of anhydrous ferrocyanide of potassium to one hundred of galena, the loss of lead diminishes to from two to two and a half per cent., probably in consequence of the easy fusibility of the mixture and the extremely fine division of the iron in the ferrocyanide of potassium. With antimonial galena this process is not applicable, as the antimony is reduced and goes into the lead. Cyanide of potassium alone gives, by reason of the greater quantity of metallic sulphide which it retains, a smaller product of lead.

### *Schemnitz Lead Assay.*

One centner of well roasted powder is mixed with two centner of black flux (of one and three-quarter parts saltpetre and two parts argol), and six to eight centner of borax, and covered with a layer of chloride of sodium.

## DETERMINATION OF LEAD BY MEANS OF STANDARD SOLUTIONS.

### 1. FLORES DUMONTÉ'S METHOD.

*Determination of Lead by means of Standard Solutions.*—This process is due to M. Flores Dumonté, and may be thus described:—This mode of analysis is analogous to that proposed by Pelouze for the determination of copper; advantage is taken of the fact that oxide of lead is soluble in caustic potash in the same manner that oxide of copper is soluble in ammonia; and from either solution the respective metal is precipitated by means of a standard solution of sulphide of sodium.

The solution of sulphide of sodium may be conveniently made by dissolving one ounce of sulphide of sodium in one quart of water, and determining how much of it is necessary to precipitate twenty grains of lead. To this end weigh off twenty grains of lead, dissolve them in nitric acid, dilute with water, add excess of caustic potash until the oxide of lead first thrown down is completely dissolved. The solution must now be heated to ebullition, and the sulphide of sodium gradually added from the bürette: at each addition a black precipitate of sulphide of lead falls. The liquid is then boiled for a short time, by which means it brightens; more sulphide of sodium is then added, and the whole again boiled, and these operations alternately continued until no further coloration or blackening is produced by the last drop of sulphide. The number of divisions used is then read off, and the calculation made as at page 356, substituting lead for copper.

Having thus standardised the solution of sulphide of sodium, the assay of a sample of ore may be thus made:—If the ore belong to the first class, dissolve it in dilute nitric acid and evaporate to dryness; to the dry mass add excess of caustic potash solution, and boil; after about a quarter of an hour's ebullition, filter, and throw down the lead as directed with the standard solution, from the amount used calculate the quantity of lead present; if the ore be of the second or third class, treat with strong nitric acid and carbonate of soda as already directed. The carbonate of lead so produced may be dissolved in either nitric or acetic acid, and to the solution thus obtained add caustic potash, &c.

### 2. SCHWARTZ'S METHOD.

Some years ago Dr. H. Schwartz published a process for the volumetric estimation of lead, which consisted in precipitating a lead solution (acidulated with nitric acid) by means of an excess of bichromate of potash; the precipitate when subsided had to be washed and filtered, and precipitate and filter placed in a freshly prepared standard solution of protochloride of iron. Decomposition took place, the chromic acid was reduced to the state of oxide, and the lead converted into, and dissolved as, chloride. When filtered and washed, the remaining undecomposed protochloride of iron was estimated by permanganate of potash, and from the difference between the remaining and original amount of iron the quantity of chromic acid was calculated, and in this way the amount of lead ascertained. This process, while it gives accurate results, requires, like that devised by Hempel, two filtrations and washings.

The present process is a more direct one. Dissolve 14·730 grammes of pure bichromate of potash in sufficient water to form one litre. One cubic centimetre of this solution precipitates 0·0207 gramme of lead.

In the estimation of pure lead a certain quantity of it should be dissolved in a minimum of nitric acid, the solution diluted with water, carefully neutralised with ammonia or carbonate of soda, and excess of acetate of soda added, and the solution precipitated by the bichromate of potash solution. When the precipitation approaches its end, or when the precipitate commences readily to subside, some drops of a neutral solution of nitrate of silver are deposited on a porcelain plate, and the chromate of potash solution only added by two or three drops at a time to the liquid under examination; after each addition the whole is well stirred, allowed to subside, and a drop of the clear supernatant liquor added to one of the drops of the silver solution. As soon as the bichromate of potash is in excess, the two drops form a red colour, while the precipitated chromate of lead has no effect on the silver test, but simply floats on the top as a yellow precipitate. Should the solution assume a yellow colour before the silver reaction has commenced, it would indicate that not sufficient acetate of soda had been added in the first instance, and it would be necessary to add this now, and also a cubic centimetre of a normal lead solution, containing 0·0207 of lead as nitrate.

The slight turbidity which first takes place soon goes off, and the operation may be proceeded with as before. One cubic centimetre must naturally, in such instance, be deducted from the amount of chrome solution, on account of the extra addition of lead. Experiments made with 0·6975 gramme of the purest lead of Tarnowitz gave the following results:—They required 33·7 cubic centimetres of bichromate of potash solution; when the silver reaction appeared, as it is always necessary to have a slight excess of bichromate of potash, we may assume that 33·6 cubic centimetres were only requisite for the precipitation of the lead. 33·6 multiplied by 0·0207 (grammes of lead)=0·6955 grammes, or 99·72 per cent., showing that it was nearly pure lead.

0·399 grammes of well-dried nitrate of lead required also 12·0 cubic centimetres of bichromate of potash, indicating 0·2484 grammes of lead, or 62·29 per cent. According to calculation, nitrate of lead should contain 62·54 per cent. of lead.

0·385 grammes of crystallised acetate of lead required 10·2 cubic centimetres of chrome solution = to 0·211 grammes, or 54·84 per cent. of lead, while according to the formula $PbO\bar{A}+3HO$ it should have been 54·61 per cent.

Of all foreign metals bismuth alone seems to interfere with the reaction, and behave very like lead with chromic acid, and if present it requires a more suitable mode of proceeding. Tin and antimony are converted into insoluble oxides during the solution of the lead in nitric acid, while arsenious acid offers no difficulties, but if desired it may be separated from the lead as sulphide with sulphide of ammonium. Arseniate of lead is insoluble in an acetic solution, and only partly decomposed by bichromate of potash, and the removal as sulphide in such instance becomes necessary. Gold and platinum are insoluble in nitric acid.

The presence of silver is of no great importance; during the operation the lead is first thrown down as a yellow precipitate, and afterwards the precipitation of the silver takes place, giving the red reaction similar to the silver test always resorted to. It may, however, be separated from the lead solution by means of chloride of sodium, and the chloride of silver either filtered off, or in case not too much chloride of sodium has been used, left in the solution, and the lead estimated as usual. Chloride of lead is tolerably soluble in hot water, and chromate of lead is not decomposed by chloride of sodium; this, however, is the case with chromate of silver.

The higher oxide of mercury is not precipitated by bichromate of potash, not even in an acetic solution, while the lower oxide is; and, as it is difficult to peroxidise all the mercury when united with lead, even by long-continued boiling in nitric acid, it becomes necessary to evaporate and calcine the residue till all the mercury is volatilised. To obviate the formation of red lead, the calcined residue has to be moistened with a few drops of oxalic acid, and again dried and carefully calcined and dissolved in acetic acid; after this, the lead may be estimated as usual. To avoid the above calcinations, the mercury may be precipitated from the nitric acid solution by means of hydrochloric acid, and the calomel boiled till it is converted into the higher chloride.

Copper, cadmium, zinc, iron, and cobalt do not in the least interfere with the reaction, provided the iron is peroxidised. Of the different acids, hydrochloric acid somewhat disturbs the last silver reaction, but by using larger drops, and allowing the reaction of chloride of silver to go off, we obtain the usual chromate of silver reaction.

Sulphate of lead has first to be converted into the state of carbonate, by boiling with carbonate of soda, when it may be dissolved in acetic acid. Phosphate and arsenite of lead, or other lead salts insoluble in acetic acid, may be dissolved in nitric acid, and estimated according to my older method.

### 3. HEMPEL'S METHOD (MODIFIED).

Mr. Sutton thus describes this method:—The lead solution, which must contain no other body precipitable by oxalic acid, is put into a 300 cc flask, and a measured quantity of normal oxalic acid added in excess; ammonia is then added to slight predominance, the flask filled to the mark with water, shaken, and put aside to settle; 100 cc of the clear liquid may then be taken, acidified with sulphuric acid, and titrated for the excess of oxalic acid with permanganate; the amount so found multiplied by 3 and deducted from that originally added will give the quantity combined with the lead.

Where the nature of the filtrate is such that permanganate cannot be used for titration, the precipitate must be collected, well washed, dissolved in dilute nitric acid, sulphuric acid added, and titrated with permanganate.

In neither case are the results absolutely accurate, owing to the slight solubility of the precipitate, but with careful manipulation the error need not exceed 1 per cent.

The following investigations of M. Levol will be found of interest. The author has investigated the subject of the quantitative determination of lead. He admits that the precise estimation of lead, though presenting no serious difficulties, nevertheless demands precautions sufficiently minute.

*The estimation of lead in the state of sulphate*, by means of sulphuric acid and evaporating to dryness, insures accuracy, but the process requires contant attention. Towards the end of the analysis the evaporation exposes it to loss by projection; moreover, if the liquids contain iron, the sulphate of lead is often contaminated with the slightly soluble ferric sulphate. The solubility of sulphate of lead, even in water, is well known, as the following experiment shows:—Precipitate one equivalent of nitrate of lead by two equivalents of sulphuric acid diluted largely with water; then wash during several days, and long after the washings have ceased to redden litmus-paper, they will still become slightly turbid by nitrate of baryta and hydrosulphate of ammonia.

*The use of soluble sulphates*, suggested by various authors, is not to be recommended, as will be shown.

M. Levol's opinion was, that the principal inconvenience arose from the incomplete insolubility of sulphate of lead, and that, consequently, the employment of alkaline sulphates would produce but imperfect results. He was then much surprised to find, under such circumstances that the fact could not escape notice, an overweight in precipitating lead by sulphate of potash. If, in fact, liquids much charged with nitrate of lead and sulphate of potash or soda in excess are put in contact, precipitates are obtained, the weight of which considerably exceeds that of the sulphate of lead, corresponding to the weight of nitrate, and it is with difficulty that they are reduced to this weight by washing.

These are some of the results:—5·1775 grammes (= 1 equivalent) of nitrate of lead, added to 5·455 grammes (=2 equivalents) of sulphate of potash, yielded a precipitate which simply drained, then dried at 110° and weighed on the filter, yielded 7·355 grammes, instead of 4·739 grammes. This precipitate was fusible, and to separate from it the sulphate it contained, no less than ninety washings in cold water were required.

An experiment made with nitrate of lead and sulphate of soda yielded 5·325 grammes of dried precipitate, instead of 4·739 grammes; and to extract from it the sulphate of soda thirty-six washings in cold water were necessary.

It seems evident that in both cases there must have been a formation of double sulphates, while that of potash appears to have been by far the most stable, the overweight of sulphate of potash being 2·616 grammes. (The formula $PbO,SO_3 + KO,SO_3$ would require 2·686 grms.) With the sulphate of soda, the overweight = 0·586 gramme, which represents about one-third of the equivalent. The formula $PbO,SO_3 + NaO,SO_3$ requires 1·704 grammes.

After prolonged washing the sulphate of lead precipitated by sulphate of soda weighed 4·640 grammes, the loss by washing having been 0·099 gramme.

The previous precipitate = 4·585 grammes.
Loss by washings = 0·154 „

Independently of less stability, this difference seems to indicate greater solubility of the double sulphate of lead and soda, which is further rendered apparent by testing the washings by means of baryta and hydrosulphate of ammonia (apart from the solubility proper to the sulphate of lead, which was taken into account in this instance), by comparing with it a third washing of sulphate of lead, formed by sulphuric acid and nitrate of lead, equivalent to equivalent.

It appears, then, that there are formed by the wet way, under certain conditions, double sulphates of lead and potash or soda.

On the whole, then, experience shows that alkaline sulphates should not be applied to the estimation of lead in the state of sulphate, by weighing the precipitate, partly because of the danger about to be described, and partly because of the fear of loss of sulphate of lead, by the numerous washings necessitated by the decomposition of the double salts by water.

**Determination of Lead in the State of Carbonate.**—In face of the difficulties to be encountered in estimating lead with great precision, it seems highly to be recommended that it should be determined in the state of carbonate: for that purpose ordinary carbonate of ammonia, to which is added caustic ammonia, is used. The object of this addition is to avoid the employment of too large a volume of solution of carbonate of ammonia, a salt not very soluble in water. Ammonia forms, with nitrate of lead for instance, a very incomplete precipitate. It would not, then, be prudent to divide the operation into two—that is to say, by employing ammonia first to saturate the liquid—and consequently it should not be poured in until it has been charged with carbonate of ammonia, which it dissolves abundantly and easily. The precipitate separates perfectly from the liquid, is easily collected and dried on a filter. The deposition of the precipitate is completed in about twenty-four hours, especially under the influence of gentle heat. According to the writer's experience, two or three thousandths of lead can be estimated by this process.

The precipitate, which is anhydrous, $PbO,CO_2$, is deposited on a small double filter, each one of the same weight.

If, as frequently happens, in analysing metallic substances, the colour, which should be pure white, is yellowish, it is owing to the presence of iron, which is easily got rid of by washing the filter after, weighing, with water acidulated with sulphuric acid.

If there is reason to suspect the presence of bismuth, treat a small quantity of the weighed precipitate with a little nitric acid. A few drops of iodide of potassium in the liquid will detect the presence of bismuth by the formation of a brown precipitate, or yellow-brown if there is bismuth and lead. The latter metal, when present alone, gives a pure yellow precipitate.

**Determination of Lead by Oxalic Acid.**—In estimating lead by carbonate of ammonia, in presence of an excess of ammonia, two or three thousandths of this metal can be determined. By operating, *under the same conditions*, with oxalic acid, it has been found impossible to determine it to less than 1 per cent. Writers have, indeed, observed, that the precipitation of lead by oxalic acid should be effected in neutral liquids; but this necessity but ill agrees with the most ordinary instances of the analysis of metallic substances, where the presence of an excess of ammonia is indispensable for maintaining in solution certain substances from which the lead should be separated.

## BLOWPIPE REACTIONS OF LEAD.

When plumbiferous compounds which are met with in nature, and furnace products, are treated on charcoal, in the oxidising flame, they give a sublimate which is very easily recognised. Other easily volatilised metals, which may be in combination with the lead, either fume away entirely, or else deposit an oxide upon the support. The oxide of lead sublimate, which is dark lemon-yellow while hot, and sulphur-yellow when cold, deposits nearer to the assay than the sublimates of some other metallic oxides, namely, those of tellurium, selenium, antimony, and arsenic, and is by this means distinguished. Should zinc also be an ingredient, the sublimate of oxide of lead will probably be contaminated with a quantity of the oxide of this metal, but the sulphur-yellow colour of the lead deposit cannot be mistaken, when the assay has perfectly cooled.

**Oxide of Lead.**—*Alone*, minium blackens when heated, and is transformed into the yellow oxide. It forms by fusion a fine orange glass, which is reduced with effervescence on charcoal.

*With borax* it fuses readily on the platinum wire, and gives a transparent glass, which, when saturated and hot, is yellowish, but which becomes colourless on cooling. It is reduced on charcoal.

*With microcosmic salt* it readily fuses into a transparent and colourless glass.

*With soda*, oxide of lead readily fuses on the platinum wire, forming a transparent glass, which becomes yellowish and opaque by cooling. Its reduction takes place instantaneously on charcoal.

## ORES OF LEAD.

**Sulphide of Lead, Galena.**—*Alone*, on charcoal, it does not fuse until after disengagement of sulphur; globules of lead then form on the surface, and finally a bead of lead is obtained. By cupelling this, the presence of silver may be ascertained. After cupellation, the bone-ash indicates by its colour whether the lead were pure or not; if it were, when cold the cupel would be pure yellow; copper renders it green, and iron brown or blackish.

*In the tube*, galena gives off sulphur, and a white sublimate of sulphate of lead.

**Oxide of Lead.**—Its action with fluxes has been already shown.

**Sulphate of Lead** decrepitates, and fuses on charcoal in the outer flame into a transparent bead, which becomes milky by cooling. In the reducing flame it effervesces, giving a button of lead.

**Carbonate of Lead** behaves like oxide of lead.

**Phosphate of Lead.**—*Alone*, on charcoal, it fuses, the bead crystallising as it cools. The crystals have large facets, and a pearly whiteness.

With fluxes it behaves like oxide of lead.

# CHAPTER XI.

## THE ASSAY OF TIN.

THIS metal is always found by the assayer in the state of oxide.

*Oxide of Tin* ($SnO_2$).—The appearance of this mineral gives no indication, excepting to an experienced eye, that metallic matter enters largely into its composition; yet its great density would lead one to suppose such to be the case. Its colour varies from limpid yellowish white to brownish black and opaque, passing from one to the other by all intermediate shades. It usually possesses a peculiar kind of lustre which cannot be readily described, but once seen can scarcely be mistaken. It occurs crystallised in square prisms, terminated by more or less complicated pyramids. These crystals, derived from the octahedron, are often macled or hemitropic, so that they often possess re-entrant angles, which is to a certain extent characteristic. The principal varieties are the following:—

1. *Crystallised Oxide of Tin* is found in more or less voluminous crystals of the colour and form as above.
2. *Disseminated Oxide of Tin.*—This variety occurs in grains of various sizes, sometimes so small as not to be visible to the naked eye. It is found in the primitive rocks.
3. *Sandy Oxide of Tin* forms pulverulent masses often of great extent; in appearance it is merely a brown sand.
4. *Concretionary Oxide of Tin, Wood Tin.*—This variety occurs in small mamellated masses, the fibrous texture of which resembles that of wood: hence the name.

The following is an analysis of a sample of oxide of tin from Cornwall:—

| | |
|---|---|
| Tin | 77·50 |
| Oxygen | 21·40 |
| Iron | ·25 |
| Silica | ·75 |

*Assay of Pure Oxide of Tin.*—Pure oxide of tin may be very readily assayed in the following manner:—Weigh off 400 grains, place them in either a black-lead or charcoal-lined crucible, cement on a cover by means of Stourbridge clay, and subject to the fire. The heat should for the first quarter of an hour be a dull red, after which it may be raised to a full bright red for ten minutes, and the crucible removed with care so as not to agitate or disturb the contents; tapping in this case must not be resorted to. When the crucible is cold, remove the cover, and a button of pure tin will result: this weighed and divided by four gives the percentage. If the operation has not been carefully conducted, it sometimes happens the tin is not in one button, but disseminated in globules either on the charcoal lining or on the sides of the black-lead pot; in this case the charcoal on the one hand, or the black-lead crucible on the other, must be pulverised in the mortar and passed through a sieve; the flattened particles of tin will be retained by the sieve, and can be collected and weighed. If any small particles escape the sieve, they may be separated from the lining or crucible by vanning.

If a charcoal or black-lead crucible be not at hand, an ordinary clay pot may be used, but not so successfully, excepting under certain circumstances to be hereafter described. Indeed, in Cornwall the ordinary mode of conducting this assay is in a naked crucible, thus: About 2 ounces of the ore are mixed with a small quantity of culm, and projected into a red-hot crucible. If the ore seems to fuse or work sluggishly, a little fluor-spar is added, and after about a quarter of an hour's fusing at a good high temperature, the reduced and fused tin is poured into a small ingot mould, and the slag examined for metal by pounding and vanning. This method never gives the whole of the metal. To effect this, without fear of mischance in the assay sometimes occurring as already described, with both black-lead and charcoal lined crucibles, it may be thus conducted; always supposing the oxide to be pure, or nearly so, or at least containing little or no siliceous matter.

To 400 grains of ore add 100 grains of argol, 300 grains of carbonate of soda, and 50 grains of lime; mix well together, place in a crucible which the mixture half fills, cover with a small quantity of carbonate of soda and 200 grains of borax. Place the whole in the furnace with the necessary precautions, raise the heat very gently, and keep it at or below a dull red heat for at least twenty minutes; then gradually increase until the whole flows freely. Remove the crucible, tap it as for copper assay, and allow to cool. When cold, break it, and a button of pure metallic tin will be found at the bottom, and a flux perfectly free from globules and containing no tin.

There is yet another process, which is more easy of execution; but the reagent employed is more expensive, not so readily obtainable, and more difficult to keep without decomposing than any of the substances above employed. The reagent now to be discussed has been introduced to the notice of the student, in another part of this volume, as a blowpipe flux, and in the assay of copper ores by standard solutions, as cyanide of potassium. This is the most effective reducing flux for tin ores yet known. It acts by absorbing oxygen to form a compound known as cyanate of potash: thus—

$$SnO_2 + KCy = Sn + KO,CyO.$$

The assay, by means of this substance, may be made in ten minutes.

This method of estimating the value of tin-stone has been frequently practised by the writer during the last nine years, and has uniformly furnished correct results with but little expenditure of time and labour. The method of operating is as follows:—The sample having been carefully selected, is first crushed by the hammer in a steel mortar, and then further reduced to powder in an agate mortar. 100 grains is a convenient quantity to be taken for analysis, and it is always advisable to make two independent experiments upon the same sample of ore, with the view of having a control, and the highest result obtained is that upon which to place reliance, since the error must always be on the side of loss rather than excess. A couple of small Hessian crucibles, of about 3 oz. capacity, are prepared in the first instance by ramming into the bottom of them a small charge of powdered cyanide of potassium sufficient to form a layer of about half an inch in depth; the weighed quantities of tin ore are then intimately mixed with from four to five times their weight of the powdered cyanide, and the mortar rinsed with a small quantity of the pure flux, which is laid upon the top of the mixture. The crucibles are then heated in a moderate fire, or over a gas-blowpipe, and kept for the space of ten minutes at a steady fusion; they are then removed, gently tapped to facilitate the formation of a single button, and allowed to cool. Upon breaking the crucibles, the reduced metal should present an almost silvery lustre, with a clean upper layer of melted flux. It is advisable to dissolve the latter in water, in order to be certain as to the absence of any trace of reduced metal or heavy particles of the original ore. There is always contained in the commercial cyanide a sufficient quantity of alkaline carbonate to secure the perfect fusion of the silicious gangue and other like impurities in the tin ore, but the operator should assure himself of the absence of copper and lead in the ore, either by preliminary treatment with hydrochloric acid, in which tin-stone is absolute insoluble, or by testing the button of reduced tin after hammering or rolling for such metallic admixture. We have usually found a minute trace of iron, and sometimes gold, in the melted buttons, but not so much as to add appreciably to their weight.

When worked with ordinary care, this process may be relied upon as giving numbers true to within ½ per cent., and we do not know any other method which exceeds this in accuracy and rapidity of execution. The following are a few analytical results taken at random from a number of ores assayed in this manner:—

| | Tin per cent. I. | Tin per cent. II. |
|---|---|---|
| Sample No. 1 | 45·6 | 45·8 |
| „ No. 2 | 57·2 | 57·6 |
| „ No. 3 | 68·4 | 68·7 |

Dr. Clemens Winkler mentions the well-known difficulty of obtaining all the tin in one button in a dry assay by the ordinary process, and the error of 5 or 10 per cent. which arises. To avoid this loss, he suggests the addition of copper for the purpose of collecting together the tin, and states that he has obtained tolerably accurate results by the following process:—

The ore is finely pulverised and roasted, first by itself and then once or twice with charcoal or coke, to remove sulphur, arsenic, and antimony. The residue is then digested for a quarter or half an hour with hot hydrochloric acid, and afterwards well washed with hot water. Iron, manganese, and copper, he states, are more completely removed by fusion with bisulphate of potash, and then treating with hydrochloric acid, and washing with water. Tungstic acid, if present, will now be removed by digesting with caustic potash or ammonia.

The oxide of tin, silica, &c., remaining is now mixed in a crucible with an equal weight of oxide of copper, and two or three parts of flux, consisting of two parts anhydrous carbonate of soda, one part white flour, and a quarter part borax glass. The whole is then covered with a layer of common salt, upon which a piece of charcoal is laid. The crucible is then heated first to a red and then to a dull white heat for an hour after, which a button containing the whole of the reduced tin and copper will be found at the bottom.

As pure oxide of copper may not be obtainable, the author recommends that a portion of every sample should be separately assayed. The weight of the tin will be found by subtracting the weight of the copper from that of the button.

*Assay of Oxide of Tin mixed with Silica.*—Although oxide of tin is completely reducible by charcoal or other carbonaceous matter, yet it has such an affinity for silica, that whenever that substance is present, the metal cannot be wholly reduced, excepting at the highest temperature of a wind furnace. The following experiments will show the influence of silica on the return of tin in an assay of oxide of that metal with black flux:—

| | | | | | |
|---|---|---|---|---|---|
| Ore | 100 | 100 | 100 | 100 | 100 |
| Quartz | 25 | 66 | 100 | 150 | 300 |

The first gave 52 per cent. of tin; the second, 43 per cent.; the third, 28 per cent.; the fourth, 10 per cent.; and the last nothing.

The slags also produced in the treatment of tin ores in the large way give no return with black flux. This mode of assay, however, has been recommended by some, but from the foregone experiments, is proved to be perfectly fallacious: that is, unless the quantity of silica present be very small in comparison to the amount of oxide of tin; and even when the latter is present in four times the quantity of the silica, as in experiment No. 1, a loss of 20 per cent. of tin is sustained.

*Assay of Tin Ores containing Silica and Tin Slags.*—It having just been shown how injuriously the presence of silica influences the produce of tin, both in ores and slags, other methods of assay than those just described must be adopted for such substances. These will be now detailed.

Tin ores containing silica may be treated by two methods: in the first the silica must be carefully separated by vanning; if the ore be well pulverised this is the best and most expeditious method. In conducting this assay take 400 or more grains of the pulverised ore according to its richness (if poor, as much as 2,000 grains may be taken), van in carefully, dry the enriched product, which will, if the operation has been properly conducted, be nearly pure oxide of tin, and assay it as already described for ores containing no silica. The other process of assay may be thus conducted, and is dependent upon the fact that iron displaces tin in its combination with silica: thus, if a compound of oxide of tin and silica be heated to whiteness with metallic iron, a portion of the iron oxidises and replaces the oxide of tin, which was previously in combination with the silica as a silicate of tin, and metallic tin and silicate of iron result, the tin so reduced combining with any metallic iron that may be in excess, and the button thus obtained is an alloy of tin and iron, whilst the slag is entirely deprived of tin.

In this kind of assay mix 400 grains of the silicated oxide of tin with 200 grains of oxide of iron (either pulverised hæmatite or forge-scales will answer this purpose), 100 grains of pounded fluor-spar, and 100 grains of charcoal powder; place the mixture in a crucible, and cover with a lid, gradually heat to dull redness, and keep at that temperature for half an hour, then heat to whiteness for another half hour, and remove the crucible from the furnace, allow to cool, and break. The button so obtained is to be treated in the humid way, as hereafter described.

The assay of tin slags is conducted in the same manner, or simply by mixing the pulverised slag with 20 per cent. of iron filings, and fusing.

*Assay of Tin Ores containing Arsenic, Sulphur, and Tungsten (Wolfram).*—In the assay of such ores it is necessary to remove arsenic, sulphur, and tungsten, before attempting to obtain the tin in a pure state by dry assay. Ores of tin which contain either one or all of these substances are most common: hence this mode of treatment will be generally required.

Most assayers usually submit the ore to the same mode of treatment which it undergoes on the large scale by calcination, or rather roasting, by which the greater part of the arsenical and pyritic matter is removed; this process fails, however, to remove the whole of these substances, and does not at all affect the tungsten. The following process, adopted by the author, is therefore preferable, and is founded on the fact that arsenical and other pyrites, as well as tungstate of iron (wolfram usually accompanying tin ores), are completely decomposed by nitro-hydrochloric acid (*aqua regia*) at the boiling temperature, the oxide of tin alone not being affected:—Take 400 grains or more of the impure tin sample, place them in a flask, and add 1½ ounce of hydrochloric acid, and ½ an ounce of nitric acid, heat gently for about half an hour, and then boil until the greater part of the mixed acids have evaporated; the sulphur and arsenic will by this time be converted into sulphuric and arsenic acid, and the wolfram completely decomposed, its iron and manganese having become soluble, and its tungstic acid remaining in the insoluble state with the oxide of tin and any silica that may be present. Allow the flask and contents to cool, add water, allow to settle, and decant, and so on until the water passes off tasteless. The insoluble matter in the flask is now oxide of tin, silica, and tungstic acid; to remove the latter, digest for an hour at a very gentle heat with one ounce of solution of caustic ammonia, with occasional agitation; add water, and van the remainder to separate silica; nothing remains now but oxide of tin with perhaps a little silica: this is now to be dried and assayed as directed for ores containing little or no silica.

If only an approximative assay be needed, it may be accomplished after this treatment by taking the specific gravity of the remaining oxide, so that all ores of tin may be thus roughly assayed, it being premised that the above operation has been so carefully performed that nothing but oxide of tin and silica remain. The specific gravity of the thus purified ore is to be taken. All now that is necessary to be known is the specific gravity of oxide of tin, its percentage of pure tin, and the specific gravity of silica, and at simple calculation gives the result. The following is the formula:—

Let $a$ represent the specific gravity of oxide of tin.
„ $b$ „ „ „ silica.
„ $c$ „ „ „ the mixture left after treatment with acid, &c.
„ $w$ „ weight of rough oxide of tin or mixture left after treatment with acid, &c.
„ $x$ „ „ oxide of tin.
„ $y$ „ „ silica.

$$\text{Then } x = \frac{a\,(c-b)}{c\,(a-b)}\,w;$$

$$\text{And } y = \frac{b\,(a-c)}{c\,(a-b)}\,w.$$

Or in arithmetical form thus,—

1. From the specific gravity of the rough oxide of tin (mixture of oxide of tin and silica) deduct the specific gravity of the silica.
2. Multiply the remainder by the specific gravity of the oxide of tin.
3. Multiply the weight of the rough oxide of tin by the last product, which will make a second product which may be called P.
4. From the specific gravity of oxide of tin deduct the specific gravity of silica.
5. Multiply the difference by the specific gravity of the rough oxide of tin.
6. Take this product for a divisor to divide the above product P: the quotient will be the weight of pure oxide of tin in the rough oxide, and the quantity of metal can now be readily calculated.

The following is an assay worked out in this manner:—

400 grains of the ore are treated with nitro-hydrochloric acid and ammonia as above described, washed and dried. Suppose the dried matter weighed 250 grains. The 250 grains thus obtained are placed in the specific gravity bottle, and the specific gravity is found to be 5·4.

Specific gravity of tin oxide (approximate) . . . 6·9
„ silica „ . . . 2·6

$$\overset{\substack{\text{Sp. Gr.}\\ \text{Rough Oxide}}}{5{\cdot}4} - \overset{\substack{\text{Sp. Gr.}\\ \text{Silica}}}{2{\cdot}6} = 2{\cdot}8$$

$$2{\cdot}8 \times \overset{\substack{\text{Sp. Gr.}\\ \text{Pure Oxide}}}{6{\cdot}9} = 19{\cdot}32$$

$$\overset{\substack{\text{Weight of}\\ \text{Rough Oxide}}}{250} \times 19{\cdot}32 = \overset{\text{P}}{4830}$$

$$\overset{\substack{\text{Sp. Gr.}\\ \text{Pure Oxide}}}{6{\cdot}9} - \overset{\substack{\text{Sp. Gr.}\\ \text{Silica}}}{2{\cdot}6} = 4{\cdot}3$$

$$4{\cdot}3 \times \overset{\substack{\text{Sp. Gr.}\\ \text{Rough Oxide}}}{5{\cdot}4} = 23{\cdot}22$$

$$\frac{4830}{23{\cdot}22} = 208{\cdot}4$$

208·4 grains is therefore the weight of pure oxide in the 400 grains of ore.

Now oxide of tin contains 78·61 parts of pure tin, and a

$$\frac{208{\cdot}4 \times 78{\cdot}61}{100} = 163{\cdot}72$$

So that 400 grains of rough tin ore contain 163·72 grains of pure tin, and

$$\frac{163{\cdot}72}{4} = 40{\cdot}93.$$

The rough sample first operated on contains, therefore, 40·93 per cent. of metallic tin.

*Estimation of Tin by the Humid Method.*—There are several methods of effecting this analysis, the chief difficulty being found in the intractable nature of the oxide of tin, it resisting the action of all acids. This, however, may be overcome as first shown by Klaproth, who found that very finely levigated oxide of tin was soluble in hydrochloric acid after a prolonged fusion with caustic potash: the following is his process:—

Fifty grains of the tin ore, reduced to the most minute state of division by levigation or otherwise, is mixed with four times its weight of caustic potash. The best mode of mixing is to place the caustic potash in a silver crucible, add its own weight of water, and apply a gentle heat until the potash is dissolved; then stir in tin ore, and gradually evaporate to dryness, stirring all the time to prevent loss by spitting, as in the analysis of iron stone: when thoroughly dry, enclose the silver crucible in one of clay, and submit the whole to a dull red heat for at least half an hour: rather more than less renders the perfect solution of the oxide of tin more certain. When cold, act on the contents of the crucible with dilute hydrochloric acid, transfer the liquid and any undissolved matter to a flask, add some strong hydrochloric acid, and boil for half an hour. If at the end of this time any of the tin ore remains unacted on, it must be separated by decantation or otherwise from the solution, dried, again fused with potash, and then treated with hydrochloric acid, in which it will now be found totally soluble. This second operation will not be needed if care has been taken to reduce the ore to the finest possible state of division at first. The solution, however obtained, is to be evaporated to dryness, and when cold treated with a small quantity of hydrochloric acid, allowed to stand for half an hour, then water added, boiled and filtered: the whole of the tin will pass through in solution as chloride of tin, and any silica or tungstic acid that may be present will remain in the filter. If the ore contained copper, lead, and iron, these metals will also be in solution—at all events, the lead partially so; but if the ore had, previously to its fusion with caustic potash, been treated with *aqua regia*, as already described, then it will contain tin alone. It is always better thus to separate foreign matters before attempting the solution of the tin, as the after process is thereby simplified.

Supposing, however, that the rough ore had been submitted to fusion with potash and then dissolved, the solution must be thus treated:—A bar of zinc must be placed in the solution, which will in course of time precipitate tin, copper, and lead; when all the metals are thus thrown down the zinc is washed and removed, the precipitated metals well washed and dried. To the dried metals strong nitric acid is now to be added, the mass gently heated, and then evaporated to dryness: when cold, it is moistened with dilute nitric acid, water added, and the whole filtered. Lead and copper will pass through the filter as soluble nitrates, and the tin will be found in the filter as insoluble peroxide; this is to be well washed, dried, ignited, and weighed. It contains 78·61 parts of metallic tin. The amount of tin thus obtained, when multiplied by two, will represent the percentage of the ore.

If, before the action of caustic potash, the ore had been submitted to the action of *aqua regia*, sulphuretted hydrogen may be passed through the solution of chloride of tin, when sulphide of tin will be precipitated; this is to be washed, dried, gently calcined in a platinum crucible until all smell of sulphurous acid has ceased, allowed to cool, reheated with a fragment of carbonate of ammonia, as in the case of roasting sulphide of copper, and when cold weighed as pure oxide of tin. The calculation for metal is made as above.

*Humid Analysis of the Alloy of Tin and Iron as obtained in the Treatment of Siliceous Ores and Slags.*—The alloy obtained as already directed is dissolved in boiling hydrochloric acid, diluted with water, and the solution, if necessary, filtered. To the filtered solution add a little hydrochloric acid and pass an excess of sulphuretted hydrogen through it, collect the precipitated sulphide of tin, and proceed according to the directions already given.

*Estimation of Tin by means of a Standard Solution.*—The first process to be described is due to M. Gaultier de Claubry, and may be thus performed:—The standard solution is made by dissolving 100 grains of iodine in 1 quart of proof spirit (spirit of wine having a specific gravity of ·920), and is thus standardised. Ten grains of pure tin are dissolved in excess of hydrochloric acid, the solution boiled, and allowed to cool: the burette is now filled with the solution of iodine, which is gradually added to that of the tin until the former ceases to be decolorised: as soon, therefore, as the tin solution assumes a faint yellow tinge, sufficient iodine has been added: the quantity thus found sufficient is then noted, and the amount of tin each divisionful of iodine solution is equivalent to, is calculated as for iron, copper, and the other standard solutions.

In the actual assay of tin ore by means of this solution it is necessary the whole of the tin present be reduced to the state of protochloride: this may be readily effected by boiling the solution of tin for a quarter of an hour with excess of metallic iron, and filtering. To the solution so obtained the iodine is added as above. The tin ore is dissolved by any of the methods already described.

M. Scheurer Kestner, of Thann, has devoted some attention to this subject. His process depends on the transformation of protochloride of tin into bichloride. MM. Streng and Mohr* have employed bichromate of potash, solution of iodine, and permanganate of potash as oxidising agents. The result of M. Mohr's experiments is that whichever of these bodies is employed, the proportion found varies with the quantities of water used, so that upon taking certain quantity of chemically pure tin, the trials made with permanganate of potash, or any other oxidiser, will not give the weight of tin employed, but always a less weight—a circumstance which has induced him to admit for these estimates an empirical atomic weight for tin (65), differing greatly from the actual weight (59). Even this number is allowable only when operating constantly with quantities of water equal to those which served to determine it.

M. Lenssen† estimates tin by means of the iodine solution, but he operates in a liquid containing double tartrate of potash and soda, and bicarbonate of soda in excess. The results M. Lenssen obtained by this method are satisfactory, by using the atomic weight of tin generally adopted (59). We shall see farther on why M. Lenssen's results agree.

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* Mohr: *Traité d'Analyse*, pp. 297 and 349.

† *Annalen der Chemie und Pharmacie*, vol. cxiv. p. 114.

M. Stromeyer* having recently occupied himself with the same subject, has succeeded in solving the difficulty. The solution of protochloride of tin is carefully introduced into an excess of sesquichloride of iron. The salt of iron becomes reduced to a minimum according to the following equation:—

$$Sn + 2(Fe_2Cl_3) = SnCl_2 + 4FeCl.$$

It is then estimated by permanganate, as if it were a salt of protoxide of iron. The results M. Stromeyer obtains in this way are very exact. The author adds that such a method of estimating is applicable only in the absence of copper or iron, as these two metals decompose permanganate of potassa, as well as the tin; but it may be of great use in the estimation of commercial salts of tin.

M. Scheurer Kestner has endeavoured to determine the causes of the variations observed when oxidising a solution of protochloride of tin, proximately by permanganate of potash, and whether, as M. Mohr supposes, these anomalies arise only from the presence of oxygen dissolved in the water. To make these experiments agree better, the solution of protochloride of tin mixed with the proper quantity of water has been instantaneously oxidised by permanganate, so as not to take time into consideration. The irregularities occurring during this operation are shown in the following table:—

| c. c. | | | | | | Permanganate. |
|---|---|---|---|---|---|---|
| 2 | of Sn Cl, without water, required | | | | | 34·5 |
| 2 | „ | and | 10 | c. c. water | „ | 34·5 |
| 2 | „ | „ | 20 | „ | „ | 34·5 |
| 2 | „ | „ | 50 | „ | „ | 34·5 |
| 2 | „ | „ | 100 | „ | „ | 34·0 |
| 2 | „ | „ | 200 | „ | „ | 33·0 |
| 2 | „ | „ | 300 | „ | „ | 33·0 |
| 2 | „ | „ | 400 | „ | „ | 31·0 |
| 2 | „ | „ | 500 | „ | „ | 31·0 |
| 2 | „ | „ | 600 | „ | „ | 29·0 |
| 2 | „ | „ | 700 | „ | „ | 30·0 |
| 2 | „ | „ | 800 | „ | „ | 28·5 |
| 2 | „ | „ | 900 | „ | „ | 28·0 |
| 2 | „ | „ | 1,000 | „ | „ | 28·0 |

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* *Annalen der Chemie und Pharmacie*, vol. cxvii. p. 261.

| | c. c. | | | | Permanganate. |
|---|---|---|---|---|---|
| | 2 | of Sn Cl and | 1,200 | c.c. water, required | 25·0 |
| | 2 | „ „ | 1,400 | „ „ | 23·0 |
| | 2 | „ „ | 1,600 | „ „ | 22·5 |
| | 2 | „ „ | 2,000 | „ „ | 17·0 |
| | 2 | „ „ | 3,000 | „ „ | 8·0 |
| | 2 | „ „ | 4,000 | „ „ | 10·0 |
| | 2 | „ „ | 5,000 | „ „ | 17·0 |
| | 2 | „ „ | 6,000 | „ „ | 18·0 |
| (A) | 2 | „ „ | 8,000 | „ „ | 24·0 |
| | 2 | „ „ | 10,000 | „ „ | 23·0 |
| | 2 | „ „ | 14,000 | „ „ | 25·0 |
| | 2 | „ „ | 24,000 | „ „ | 32·5 |
| (B) | 2 | „ „ | 24,000 | „ „ | 32·0 |

Leaving A and B to rest for ten minutes,—

| | | | |
|---|---|---|---|
| (A) | required | 14·0 | of permanganate. |
| (B) | „ | 25·0 | „ |

The result of these experiments is that the more the solutions are diluted, the less permanganate is necessary until the proportion becomes about 10,000 parts of water to one of tin (100 cubic centimetres of the oxidising liquid corresponding to one gramme of tin). From this point the quantities of permanganate necessary are again augmented, and when the dilution is brought to $\frac{1}{72000}$th, as much permanganate is needed as when it was only at $\frac{1}{4000}$th. The oxygen of the water then seems to have very little action on concentrated solutions, while on diluted solutions its action is very marked. At a certain degree of dilution, however, these phenomena are reversed, and instead of continuing to augment, the sensitiveness of the liquid for oxygen diminishes more and more, becoming gradually reduced to what it was in the concentrated solutions. In the preceding experiments no account has been taken of time; the oxidation always took place immediately after the protochloride was mixed with water. By allowing the very dilute protochloride to remain several minutes before adding the permanganate, a much greater absorption of the oxygen of water takes place, as shown by the experiments A and B.

If the oxygen of water is the cause of these variations, they ought not to take place when boiled water freed from air is used; and in fact they do not then take place. Water was boiled, and the temperature then lowered to 80°, the protochloride was then introduced, and the estimation proceeded with. The following results show that in operating thus the standard does not vary, whatever quantities of water are used.

| Protochloride employed | Quantities of Water | Standards found | |
|---|---|---|---|
| | | With Cold Water | With Boiled Water |
| 2 | 100 | 26 | 26 |
| 2 | 200 | 23 | 26 |
| 2 | 300 | 22 | 26 |
| 2 | 400 | 21 | 26 |
| 2 | 800 | 19 | 26 |
| 2 | 900 | 17 | 26 |
| 2 | 1,000 | 16 | 26 |
| 2 | 1,200 | 13·5 | 26 |

For studying the action of free oxygen on diluted and concentrated solutions of tin, the author passed a current of this gas into various solutions of protochloride of tin. For this purpose two flasks, communicating by a tube, are filled, one with a solution of 2 cubic centimetres of protochloride of tin in 100 cubic centimetres of water, and another with the same quantity of protochloride in 1,000 cubic centimetres of water. The oxygen current, forced to traverse the contents of the first flask before arriving at the contents of the second, during an hour, the liquids in the two flasks filtered and tested by permanganate of potash, yielded the following results:—

c.c. c.c.
1st Flask, containing 2 Sn Cl in 100 water, required 25·5 permanganate.
2nd Flask „ „ 1,000 „ „ 9·2 „
2 c. c. Sn Cl, in 1,000 of water, without oxygen current, 16·0 „

The first flask contained the strongest solution, and received the oxygen first, and absorbed only an amount of oxygen represented by 0·5 of permanganate; while the metal solution of the second flask absorbed a quantity represented by 16 − 9·2 = 6·8 of permanganate. The same experiment repeated by employing only 50 cubic centimetres of water in the first flask, and by continuing the oxygen current for two hours and a half, gave the following numbers:—

1st Flask, containing 50 c. c. of water, 2·6 permanganate.
2nd Flask „ 1,000 „ „ 3·2 „

In this case the concentrated solution of protochloride of tin did not absorb oxygen, while the diluted solution absorbed a quantity equivalent to 16 − 3·2 = 12·8 of permanganate, that is to say, nearly half the oxygen necessary to oxidise the protochloride completely.

M. Mohr's experiments prove* that the oxygen of water has not the slightest action upon salts of protoxide of iron, whilst under the same conditions the protoxide itself oxidises immediately. M. Mohr has taken advantage of this property to estimate by means of permanganate the oxygen dissolved in water. It is interesting then to observe whether there exists between protochloride and protoxide of tin a difference of affinity for oxygen, analogous to that distinguishing the salts of iron of the oxide. This analogy, in fact, exists, only the reactions are inverse; that is to say, protoxide of tin manifests no affinity for free oxygen, while, as we have seen above, the protochloride combines more or less rapidly with this gas dissolved in water.

For estimating the oxide of tin by permanganate of potash (or, what comes to the same thing, a solution of protochloride with the addition of carbonate of soda), M. Kestner made use of the ingenious method M. Péan de Saint-Giles † employed while studying the oxidising action of permanganate in alkaline liquids. This method consists, as is well known, in the joint use of permanganate of potash and protochloride of iron.

In making the following experiments a slight excess of carbonate of soda was added to the protochloride of tin, then an excess of permanganate of potash; and, lastly, ferrous chloride and acid.

Thus, the following are the standard solutions :—

| Sn Cl. employed Cubic centimètres | Quantities of water | Observed Standard | |
|---|---|---|---|
| | | With the protoxide | With the protochloride |
| 2 | 400 | 14 | 14 |
| 2 | 500 | 14 | 14 |
| 2 | 600 | 14 | 12 |
| 2 | 700 | 14 | 10 |
| 2 | 1,000 | 14 | 8 |
| 2 | 1,500 | 14 | 5·5 |
| 2 | 2,000 | 14 | 5 |
| 2 | 3,000 | 14 | 4·2 |
| 2 | 3,000 | 14 | 4 |

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* *Traité d'Analyses par Liqueurs Titrées*, p. 260.

† *Annales de Chimie et de Physique*, vol. iv. third series.

The small degree of affinity between oxide of tin and oxygen is made evident by passing a current of oxygen in a solution of protochloride of tin with carbonate of soda in excess. A solution containing 2 cubic centimetres of protochloride to 1,000 cubic centimetres of water is unaltered, even though a rapid current of oxygen is passed through it for two hours.

The oxygen of water then has no influence on protoxide of tin, and the results obtained are very regular, which explains why M. Lenssen, by using an alkaline liquid, arrived at corresponding results.

This property might be of service in estimating the oxygen dissolved in water if the oxidation of the protochloride were not so slow. It is sometimes necessary to wait very long before adding the permanganate, until all the oxygen of the water is absorbed, which is always done after some time, as may be seen by the following experiments:—

| Water | Permangante required by the Chloride of Iron | Permangante required by the Protoxide of Iron | Permangante required by the Protochloride of Tin | |
|---|---|---|---|---|
| 1,500 | 24 | 12·5 | After quarter of an hour | 15 |
| | | | After half an hour | 13 |

We have seen that M. Stromeyer, by a happy modification, has reduced the estimation of tin to that of iron. Applying the same principle, a salt of copper may be substituted for a salt of iron. An equivalent quantity of copper can thus be estimated in place of tin; and M. Mohr's as well as M. Terreil's* experiments show that copper can be very exactly estimated by permanganate of potash.†

A double decomposition takes place on protochloride of tin being added to nitrate or chloride of copper in excess;

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* *Comptes-Rendus*, vol. xlvi. p. 230.

† For the estimation of copper, variable quantities of water can be used without affecting the results:—

| Water employed | Permanganate required by a reduced Salt of Copper | | |
|---|---|---|---|
| 0 | 16·3 | | |
| 100 | 16·4 | 16·3 | 16·4 |
| 500 | 16·3 | | |
| 1,000 | 16·3 | | |
| 1,500 | 16·4 | 16·3 | 16·3 |
| 2,000 | 16·3 | | |
| 2,500 | 16·4 | 16·3 | |
| 3,000 | 16·4 | | |

a salt of suboxide of copper forms, and the tin passes to the maximum state of oxidation, according to the following equation:—

$$\mathrm{4CuO\quad 2SnCl = 2Cu_2O + SnCl_2 + Sn_2O}.$$

To estimate tin, it is, then, sufficient to transform it into protochloride, to add to it a solution of nitrate of copper slightly in excess, before diluting it with water, and to estimate the liquid obtained by permanganate of potash.

The following experiments show that, whether the operation is performed with nitrate of copper or whether we avail ourselves of oxidation in an alkaline medium, the results are the same:—

| | | |
|---|---|---|
| 10 c. c. SnCl in 1,200 c. c. of water required . | 52 | of permanganate |
| 10 „ SnCl in 1,200 c. c. of water, to which is added $\mathrm{CuO.NO_5}$, required . . . | 87 | „ |
| 10 „ $\mathrm{SnCl + NaOCO_2 + SO_3 + FeCl}$ required . | 231 | „ |
| Deducting that needed for the salt of iron . . | 144 | „ |
| Leaves the remainder . . . . . | 7 | the salt of tin. |

1·117 grains of tin, obtained by electrolysis, were dissolved in hydrochloric acid and diluted with water sufficient to make the entire volume 200 cubic centimetres. 30 cubic centimetres of this solution required 9·2 cubic centimetres of permanganate, whether the operation was performed with nitrate of copper or with an alkaline solution.

Permanganate was standardised by means of crystallised double sulphate of iron and ammonia, $\mathrm{FeO,SO_3 + NH_4O, SO_3 + HO}$.

2·918 grains of this salt required 24 cubic centimetres of permanganate. The salt containing 14·386 per cent. of iron, the 2·918 grammes of it contain 0·416865, or, rather, 1 cubic centimetre of permanganate $=$ 0·017268 of iron corresponding to 0·01818 of tin ($59\ \mathrm{Sn} = 2 \times 28 = 56\ \mathrm{Fe}$).

9·2 cubic centimetres of permanganate were required by 30 cubic centimetres of the foregoing solution of tin, making 0·16725 of tin.

Thus we have:—

| | |
|---|---|
| Tin taken . . . . | 0·16755 |
| Tin found . . . . | 0·16725 |
| Difference = | 0·00030 |

By calculating the equivalent of tin by these data, and by removing the iron from it, we obtain 59·105, which differs little from 59·00, the number generally adopted.

The result of these experiments is, that by passing an oxygen current through a concentrated solution of protochloride of tin, the oxygen is not fixed; in diluted solutions, on the contrary, the oxygen is rapidly absorbed by the stannous salt. Oxygen does not act on hydrated protoxide of tin. The variations noted in the strength found when protochloride of tin is oxidised by permanganate of potash are not observed in concentrated solutions; they take place only in presence of oxygen dissolved in water.

There are then three different processes for estimating tin by permanganate of potash:—1. To operate with water freed from air by boiling, protecting it from access of air while cooling. 2. To oxidise protoxide of tin in an alkaline medium. 3. To decompose protochloride of tin either by a salt of iron, as proposed by M. Stromeyer, or by a salt of copper.

## BLOWPIPE REACTIONS OF TIN.

Tin deposits an oxide upon charcoal which is feebly yellow, and moderately phosphorescent when hot; on cooling it is white, and almost touches the assay. It assumes, with cobalt solution, a bluish-green colour, which may be readily distinguished from that produced by zinc.

*Tin Pyrites.*—Tin is readily recognised in this mineral by exposing a small piece of it to the oxidising flame on charcoal. The assay at first exhales a sulphurous acid smell, afterwards becomes snow-white on the exterior, and a white coating is perceived on the support surrounding the specimen: this sublimate is so abundant, that the charcoal is not seen in any part between it and the metallic bead. This deposit is not expelled in either flame; in other respects, its compartment is similar to the oxide of tin.

The best method for the detection of tin in tantalites and tin slags is by reduction with soda; but in such a case it is necessary to add a small portion of borax, to dissolve the tantalic combinations, and prevent the reduction of iron. After the completion of the process, the tin is obtained by pulverisation and sifting. To be convinced that the metallic particles obtained are tin, dissolve protoxide of copper in microcosmic salt, add some of them to the flux, and then heat the whole upon charcoal in the reducing flame. If tin is present, the glass will be coloured reddish on cooling.

**OXIDES OF TIN.**—*Alone*, the protoxide, in the state of hydrate, lights and burns like tinder, becoming peroxidised. The peroxide does not fuse or undergo any change except in the reducing flame, which, if strong and long continued, entirely reduces it without the aid of any reagent. Nevertheless, this operation requires much practice and experience.

*With borax* it fuses with great difficulty and in small quantity, giving rise to a transparent and colourless glass, which remains so during cooling. The colour of the glass is not changed in the reducing flame.

*With microcosmic salt* it behaves as with borax.

*Soda* and oxide of tin combine with effervescence on the platinum wire. The result of this combination is a blebby infusible mass, which cannot be dissolved by a large quantity of borax. On charcoal it is easily reduced, and gives a grain of tin.

# CHAPTER XII.

## ASSAY OF ANTIMONY.

ANTIMONIAL substances susceptible of being assayed by the dry way are divisible into two classes.

CLASS I. In this class are comprised native antimony and all antimonial substances containing oxygen or chlorine, and but little or no sulphur.

These substances are the following:—

Native antimony, $Sb$,
Oxide of antimony, $Sb_2O_3$,
Antimonious acid, $Sb_2O_4$,
Antimonic acid, $Sb_2O_5$.

CLASS II. includes the sulphide of antimony and all antimonial ores containing much sulphur.

Sulphide of antimony, $Sb_2S_3$,
Oxysulphide of antimony, $Sb_2O_3+2Sb_2S_3$,
Haidingerite, $2Sb_2S_3+3FeS$.

### ASSAY OF ORES OF THE FIRST CLASS.

All the oxides of antimony are very readily reduced by charcoal; so that their assay presents no difficulty. The assay is conducted in precisely the same manner as that of oxide of lead; only, as antimony is much more volatile than lead, the heat must be managed with care, and the assay taken from the fire as soon as finished. When all suitable precautions are taken, the loss of antimony is not very considerable; but Berthier says it is never less than from 5 to 6 per cent. This, I think, is too high. Thus the pure protoxide gives 77 per cent. of metal, and antimonious acid 75. The reduction is readily made, without addition, in a charcoal crucible; but when the substance to be assayed is mingled with impurities, some flux must be added. It succeeds equally well with 3 parts of black flux, with 1 part of tartar, with 1 part of carbonate of soda, and 15 per cent. of charcoal, or any other equivalent reducing flux.

When the substance under assay contains oxide of iron, the latter oxide is more or less reduced, and the metallic iron alloys with the antimony.

Oxidised matters which contain but a small quantity of sulphur can also be assayed in this manner; because the sulphide gives up to black flux the small quantity of antimony which it contains, so that but little remains in the slag. The common glass of antimony produces by this method of assay 70 per cent. of antimony, and occasionally even more than that.

The ores of this class occur very seldom, and are only in rare cases subject to assaying.

## ASSAY OF ORES OF THE SECOND CLASS.

As pure sulphide of antimony (antimonium crudum) as well as metallic antimony (regulus of antimony) are mercantile substances, the assays of the ores of this class have for their object the determination of both these bodies.

### I. DETERMINATION OF THE PURE SULPHIDE OF ANTIMONY (ANTIMONIUM CRUDUM).

Sulphide of antimony is almost the only mineral from which antimonium crudum is produced. This mineral generally occurs intermixed with very refractory gangue (gneiss, quartz, limestone, etc.). Sulphide of antimony fuses readily at a low red heat, and is not changed during fusion, if atmospheric air is precluded. At a white heat it volatilises without change of composition.

The assay of sulphide of antimony is now effected by a liquation process, i.e. by heating the mineral sufficiently in order to melt the sulphide of antimony, and, by this means, to separate it from the refractory gangue. The production of sulphide of antimony on a large scale is done in the same way.

For the assaying purpose, two pots or crucibles are used, one standing in the other one, and leaving sufficient space between the two bottoms to receive the fused sulphide of antimony. The bottom of the inside crucible is furnished with holes. The mineral to be assayed is put into the inside crucible, the latter is then closed with a cover, and hermetically luted; the joints of the two crucibles are also luted. The under crucible is then put on the hearth of a furnace, enclosed with ashes or sand, in order to keep it cool, and the upper crucible, as far as it is outside of the under crucible, is covered with coal, and heated to a moderate red heat. The sulphide of antimony will then melt and collect in the under crucible, from which it may be taken out, after cooling, and weighed.

### 2. Determination of Regulus of Antimony.

This assay may be made in two ways: first, by roasting and fusing the oxidised matter with black flux; secondly, by fusing the crude ore with iron, or iron scales, with or without the addition of black flux.

The roasting of sulphide of antimony requires much care, for it is very fusible and volatile, as is also the oxide its decomposition gives rise to. The heat ought to be very low during the operation, and the substance continually stirred. When no more sulphurous acid is given off, we may feel assured that it is perfectly roasted, because no sulphate is ever formed in this operation.

The roasted sulphide is then fused with three parts of black flux, or its equivalent.

Metallic iron very readily separates all the sulphur from sulphide of antimony; but as sulphide of iron has a specific gravity near that of antimony, the separation is very difficult to manage: a strong fire must be employed when the desulphurisation is complete, to keep the whole body in full fusion, for a considerable time With these precautions two buttons are obtained, which separate very well: the one white, and in large plates, which is antimony; and the other a bronze yellow, a little brighter than the ordinary sulphide of iron, because it is mixed with a little metallic antimony. During the operation a very considerable portion of antimony is always volatilised, which, by this process, is an inconvenience impossible to avoid.

It is, nevertheless, practised in the large way in some factories; but a good result is not generally obtained. It, however, appears that when all the necessary precautions are taken, it can be employed with advantage.

The first precaution which is indispensable is, mixing with the sulphide only the precise proportion of iron necessary to effect its decomposition, which quantity amounts to about 42 per cent. of its weight. If more be placed, the antimony, having a great tendency to play the part of an electro-negative element, will combine with the surplus, and an antimonide of iron result, part of which will remain in the antimony and part in the slag.

Further, the iron ought to be in the finest possible state of division. If the masses be large, a portion of sulphide of antimony is volatilised before they can be fully attacked. In general, 63 per cent. of antimony can be extracted from sulphide by the aid of iron in the small way, but on the large scale it seems that 55 per cent. is the maximum.

Cast-iron cannot be employed instead of wrought, because sulphur has very little action in it. The desulphurisation is imperfect, and the slag adheres to the reduced metal.

One of the greatest inconveniences in separating sulphur from antimony by means of iron is the strong heat necessary to separate the slag from the metal. This might be remedied by making the slag more fusible and less heavy, by the addition of some flux, as an alkaline carbonate or sulphate.

If sulphide of antimony be fused with an alkaline carbonate and charcoal, regulus is obtained, and a slag composed of an alkaline sulphide and sulphide of antimony. If metallic iron be thrown into this slag whilst in fusion, all the antimony separates immediately, and a new slag is formed as fluid as the former, containing sulphide of iron and sulphide of the alkaline base employed. If, instead of the above process, the iron be mixed intimately with the sulphide of antimony and carbonated alkali, the result is the same—100 parts of sulphide, 42 of metallic iron, 50 of carbonate of soda mixed with one-tenth of its weight of charcoal, or 50 of black flux: give 65 to 66 of regulus; with the same proportion of iron, and only 10 of flux, only 62 per cent. can be obtained. In these two cases the fusion takes place very rapidly and without bubbling, and the slag, which is very liquid, separates readily from the metal. By employing 1 part of alkaline flux, the proportion of iron can be reduced from 25 to 30 per cent., and the product of metal is always from 65 to 66 per cent.

Hence, in making an assay of sulphide of antimony, it is always better to employ a smaller quantity of iron than is necessary to complete the desulphurisation, and make up for it by increasing the quantity of flux: then it may be insured that no excess of iron will be present.

The alkaline sulphates are decomposed into alkaline sulphides by the agency of charcoal at a slightly elevated temperature. The sulphides of the alkaline metals, by combining with the other metallic sulphides, augment their fusibility very considerably. Thus when sulphate of soda, mixed with about one-fifth of its weight of charcoal, is added to a mixture of sulphide of antimony and metallic iron, the metallic antimony separates very rapidly, and the slag almost instantly becomes perfectly fluid.

But it must be noted that the presence of an alkaline sulphide diminishes the product of regulus, unless the proportion of iron be augmented at the same time.

For instance, with

100 parts of sulphide of antimony,
42 parts of iron,
100 parts of sulphate of soda,
20 parts of charcoal,

but 22 parts of regulus were furnished; but with

100 parts of sulphide of antimony,
42 parts of iron,
10 parts of sulphate of soda,
2 parts of charcoal,

62 parts of antimony were easily obtained.

Instead of metallic iron, pure oxide of iron may be used, or any ferruginous matter whatever, provided it is rich; but it is necessary to add, at the same time, an alkaline flux and charcoal to reduce the oxide of iron.

Not less than 40 parts of iron scales can be employed for 100 of sulphide of antimony, and then, on the addition of 50 to 100 parts of carbonate of soda and 8 to 10 of charcoal, about 56 of regulus are obtained; but if with 100 parts of carbonate of soda from 14 to 15 parts of charcoal be employed, 65 parts of antimony are the result. By augmenting the proportion of scales, that of soda may be diminished. Thus, if from 56 to 60 parts of scales, 10 of soda, and 10 of charcoal be employed, 50 parts of regulus are the result; and if the proportion of soda be 50, and that of carbon 10, from 65 to 66, and even 67, parts of regulus are obtainable.

The fusion always takes place quickly, and the slags are very fluid.

When sulphide of antimony is fused with forge slag (silicate of protoxide of iron), carbonate of soda, and charcoal, a very white crystalline regulus, in large plates, is obtained; together with a bronze yellow matter, and a black, opaque, vitreous slag, shining like jet, in which the greatest portion of the alkali employed appeared to be concentrated. These three substances separate very readily from each other.

100 parts of sulphide of antimony,
80 parts of forge slag,
50 parts of carbonate of soda,
10 parts of charcoal,

produced very readily 60 parts of regulus.

The best method of assaying sulphide of antimony seems to be one in which it is mixed with four parts of cyanide of potassium, and heated very gently in a crucible. The heat required in this case is so low, and the operation is made so quickly, that none, if any, of the antimony is lost: so that this process is decidedly preferable in the way of an assay. In particular cases, however, the wet method must be had recourse to.

The sulphide of antimony is analysed by boiling with aqua regia. The residue consists of sulphur and gangue. It is to be washed and dried, then weighed and ignited. The loss will be sulphur, and the remainder is pure gangue.

Water is then added to the filtered solution, which will cause the precipitation of some of its contained antimony as oxichloride: this must be separated by filtration. The solution is then to be saturated with carbonate of potash, and a new precipitate will be formed. The solution is to be filtered, and made slighly acid; then nitrate of baryta must be added to it to separate its sulphur as sulphate of baryta, which it to be washed, dried, and weighed: its weight indicates the amount of sulphur: 116 parts are equal to 16 parts of sulphur.

The precipitate by water of oxichloride which remains on the filter is redissolved by hydrochloric acid, and its antimony separated in the metallic state by means of zinc. The precipitate formed by carbonate of potash can contain lead, copper, iron, and antimony. It must be treated by nitric acid, which dissolves everything but the antimony, which may then be estimated as antimonic acid.

It is always best, before conducting the analysis of sulphide of antimony, to affuse it with very dilute hydrochloric acid, in order to dissolve a portion of the carbonate of lime, which may form part of the gangue. As the composition of the sulphide of antimony is constant, the following process is sufficient in the assay of an antimonial ore:—

Boil the ore, after treatment with dilute hydrochloric acid, with concentrated hydrochloric acid, which dissolves only sulphide of antimony, and precipitate the metal as oxichloride by means of water.

Or, after all gangues soluble in dilute hydrochloric acid have been removed, the residue may be weighed, and then acted on by boiling hydrochloric acid, until all action ceases. The residue must be well washed with weak hydrochloric acid, dried, ignited, and weighed; the loss of weight corresponds to the percentage of pure sulphide of antimony, which contains 72·7 per cent. of metal.

The following methods of determining antimony are given by Mr. Sutton.

### 1. *Conversion of Oxide of Antimony in Alkaline Solution into Antimonic Acid by Iodine* (*Mohr, results accurate*).

The oxide of the metal, or any of its compounds, is brought into solution as tartrate by tartaric acid and water; the excess of acid neutralised by neutral carbonate of soda, then a cold saturated solution of bicarbonate of soda added in the proportion of 20 c. c. to about 0·1 Gm. of $SbO_3$; to the clear solution starch liquor and $\frac{N}{10}$ iodine are added until the blue colour appears; the colour disappears after a little time, therefore the first appearance of a permanent blue is accepted as the true measure of iodine required.

1 c. c. $\frac{N}{10}$ iodine= 0·0061 Gm Sb.

### 2. *Distillation of Ter- or Pentasulphide of Antimony with Hydrochloric Acid and Titration of the evolved Sulphuretted Hydrogen* (*Schneider, results accurate*).

When either of the sulphides of antimony are heated with hydrochloric acid in Bunsen's, Fresenius', or Mohr's distilling apparatus, for every 1 eq. of antimony present as sulphide 3 eq. of sulphuretted hydrogen are liberated. If therefore the latter be estimated, the quantity of antimony is ascertained. The process is best conducted as follows:—

The antimony to be determined is brought into the form of ter or pentasulphide (if precipitated from a hydrochloric solution, tartaric acid must be previously added, to prevent the precipitate being contaminated with chloride), which, together with the filter containing it, is put into the distilling flask with a tolerable quantity of hydrochloric acid not too concentrated. The condensing tube contains a mixture of caustic soda, or potash, with a definite quantity of $\frac{N}{10}$ arsenious acid solution, in sufficient excess to absorb all the sulphuretted hydrogen evolved. The flask is then heated to boiling, and the operation continued till all evolution of sulphuretted hydrogen has ceased; the mixture is then poured into a beaker, acidified with hydrochloric acid, to precipitate all the tersulphide of arsenic. The whole is then diluted to, say 300 c. c., and 100 c. c. taken with a pipette, neutralised with carbonate of soda, some bicarbonate added, and the titration for excess of arsenious acid performed with $\frac{N}{10}$ iodine and starch.

## BLOWPIPE REACTIONS OF ANTIMONY.

**Red and Black Sulphides of Antimony.**—*Alone*, they fuse readily on charcoal, which absorbs them and becomes covered with a black vitreous crust. After the blast has been continued for a few moments, metallic globules appear on the charcoal, which seem to be a sub-sulphide, as they do not behave like the pure metal; for they do not burn, but blacken, and become dull on the surface after cooling.

Roasted in the glass tube, much antimonious acid forms at the commencement; that which sublimes afterwards is a mixture of antimonious acid with much oxide.

**Antimony and its Oxides.**—*Metallic antimony* fuses readily on charcoal, and, when heated to redness, remains a considerable time in a state of ignition without the aid of the blowpipe, disengaging a thick white smoke. This smoke is gradually deposited on the charcoal around the metallic globule in small crystals, having a pearly lustre, and which, in course of time, cover it entirely. These crystals are oxide of antimony. Metallic antimony alone in the matrass does not sublime but at the fusing point of glass. Heated to redness in the open tube, it burns slowly, giving a white smoke, which deposits on the glass, and presenting here and there traces of crystallisation.

*Oxide of Antimony.*—*Alone*, readily fuses, and passes off as a thick white vapour. It is reduced to the metallic state on charcoal. In this operation it colours the flame greenish.

*Antimonious Acid* does not fuse, but gives off a vivid light, diminishing at the same time in bulk, and covering the charcoal with a white powder, but is not reduced.

*Antimonic Acid* whitens at the first impingement of the flame, and is converted into antimonious acid.

The oxides and acids of antimony behave alike with fluxes.

*Borax* dissolves a large quantity of antimonious acid without becoming opaque. The glass continues yellow while hot, but loses nearly all its colour on cooling. When saturated, a portion of the antimony sublimes in the metallic state.

*Microscomic salt* forms with the same acid a transparent and colourless glass. On the platinum wire, in the oxidating flame, it becomes yellow, which tint vanishes on cooling.

*With soda*, on the platinum wire, it fuses into a transparent and colourless glass, which becomes white on cooling. It is reduced on charcoal.

# CHAPTER XIII.

## THE ASSAY OF ZINC.

ALL bodies containing zinc, usually found in the assay office, may be divided into four classes :—

Class I.—Zinc ores, in which the metal exists as oxide not combined with silica :—

Earthy oxide of zinc, $ZnO$.
Manganiferous oxide of zinc, brucite, $ZnO+(MnO)^n$.
Aluminate of zinc, Gahnite, $ZnO,6Al_2O_3$.
Franklinite, $3(FeO,ZnO)+(Fe_2O_3,Mn_2O_3)$.
Anhydrous carbonate of zinc, $ZnO,CO_2$.
Hydrated carbonate of zinc, $ZnO,3HO+3ZnO,CO_2$.

Class II.—Zinc ores, in which the metal exists, as in the former class, as oxide, but partly or wholly combined with silica :—

Anhydrous silicate of zinc.
Hydrated silicate of zinc, electric calamine, $ZnO,{}_2SiO_3+ZnO,HO$.

Class III.—Zinc ores, in which the metal is partly or wholly combined with sulphur:—

Sulphide of zinc (blende, *Black Jack*), $ZnS$.
Oxysulphide of zinc. (This is rare.)
Sulphate of zinc, $ZnO,SO_3,7HO$.
Selenide of zinc, $ZnSe$. (Very rare.)

Class IV.—Alloys.

### ASSAY OF ORES OF THE FIRST CLASS.

In order to reduce the oxide of zinc contained in substances of this class, it is sufficient to mix them with charcoal and expose them to a white heat.

At the moment of reduction the zinc is in a vaporised state. Its vapours, however, are readily condensible, so that the operation may be conducted in an ordinary retort, and all the metal is deposited in the neck without the slightest loss. It seems from this that nothing is so easy, at first sight, as the assay of an oxide of zinc; but it is not so. It is very easy to reduce all the oxide, but it is not so easy to collect all the zinc; nor is it easy to condense it all in the metallic state, and in consequence to determine the precise proportion in the ore submitted to assay.

This difficulty consists, firstly, in the deposit being extended over a large surface, and it often adheres very strongly to the sides of the retort, so that it is nearly impossible to detach it; and secondly, as the neck of the retort is open, the air having access to it, brings to the state of oxide all the vapour nearest the end of the neck. The proportion of zinc oxidised is larger in proportion to the smallness of the quantity submitted to assay, and is always very considerable where no more than 200 to 400 grains are operated upon.

It is not, therefore, in the extraction of the zinc from its oxide that the assay is rendered partially uncertain, but in its collection.

The distillation of zinc requires a very high temperature, and cannot be performed in retorts of glass; those of earthenware must be employed. It is not necessary to lute these retorts when they are of good quality; and they are better thin, because they heat more rapidly, and are not so likely to crack.

After the mixture of oxide and charcoal has been introduced into the retort, it is placed in the fire. The neck ought to have adapted to it a long tube of glass, with a narrow bore, so as to collect all the zinc which may escape from the wide part of the neck of the retort. This disposition is also convenient, as it does not allow such a free access of air.

It is heated gradually until it is white inside; the zinc is reduced and volatilised, and condensed in the neck: the greater the heat, the nearer the orifice. The metal can be detached readily from the neck, if it be well black-leaded inside. It is necessary, from time to time, to observe the state of the neck, because when very narrow it is often obstructed, and, if not cleaned out with an iron rod, might cause an explosion.

When the operation is finished the apparatus is allowed to cool, the retort taken out, placed carefully on its side, and broken, in order that if any particles of zinc have condensed in its dome, they may be removed.

If the approximate proportion of metallic zinc alone be the end, all is collected and fused at a very gentle heat in a crucible with some black flux; but if the true quantity of zinc is to be estimated it must be done in a more exact manner. The deposit must be collected with all possible care; the neck must then be broken to pieces, and every piece having adhering to it either zinc or oxide must be placed on one side, and digested in hot nitric acid, which takes up those substances. If any be put in the glass tube, it must be carefully cleaned by means of acid, and the solution added to that produced by the digestion of the broken neck, and the deposit mechanically collected, in nitric acid. The solution is then evaporated gradually to dryness, and heated to redness. The nitrate, by these means, is decomposed, and transformed into oxide, four-fifths of the weight of which is equal to the quantity of metallic zinc produced in the assay.

The foregoing is the method of estimation by distillation; the following is the method of estimation by difference. Two plans of assay in this manner may be adopted: firstly, at an ordinary assay temperature; secondly, at a very high temperature, as that of an iron assay. In all cases it is necessary to commence with the expulsion of all volatile bodies the ore may contain. If water or carbonic acid alone be present, simple calcination will do; but if carbonaceous matter, roasting must be had recourse to.

When the assay is made at an ordinary assay temperature, the sample is finely pulverised, and mixed with from 15 to 20 per cent. of equally finely pulverised charcoal, and pressed into a crucible, on which is placed a cover, but not luted, and rapidly heated to whiteness. When no more zinc vapour is disengaged it is cooled, and the mixture in the pot collected. The residue ought to be pulverulent; but as it is mixed with some charcoal, it is roasted, and then weighed. It is evident that the loss represents the oxide of zinc: the charcoal added, it is true, leaves a small quantity of ash, but it is too small to be accounted for.

In making the assay in the manner described, it is to be feared that a small quantity of the oxide remains undecomposed, and that a part of the residue might adhere to the crucible, and could not be detached; and lastly, there is always a degree of uncertainty in the state of oxidation the iron the substance may contain will exist in it after roasting. No inconvenience of this nature presents itself when the assay is made at a very high temperature. This mode is the most exact of all, and leaves nothing to be desired.

The assays of zinc at a high temperature are made exactly as those of iron. They are made in a charcoal crucible, with the addition of fixed fluxes, suitable to effect the fusion of the gangues mixed with the oxide of zinc, if they be not fusible by themselves. The button is weighed; it is a compound of slag and grains of iron, which are collected and their weight ascertained, and, by the difference, that of the slag. The weight of oxygen which the iron has lost during its reduction is then added to it, and by deducting from the substance the weight of the button and the oxygen so obtained, we have the proportion of oxide of zinc reduced in the assay. On the other hand, by deducting from the weight of the slag the weight of flux added, the weight of earthy substances and irreducible oxides which were mixed with the oxide of zinc is ascertained.

These results can be shown in a tabular form, in the following manner:—

Let $m$ be the weight of the crude ore, $n$ the weight of the calcined ore, $r$ the weight of the flux added, $f$ the weight of the cast-iron, $s$ the weight of the slag, $o$ the weight of oxygen combined with the iron, calculated from the weight of metal produced, $z$ the weight of the oxide of zinc, then :—

$$\begin{array}{lr} m \text{ crude ore}=\text{calcined ore} \quad . \quad . \quad . \quad . \quad . & n \\ r \text{ fixed fluxes} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . & r \\ \hline & n+r \end{array}$$

$$\begin{array}{l}\text{Gives metal} \quad . \quad . \\ \text{Gives slag} \quad . \quad . \quad . \end{array} \left.\begin{array}{c} f \\ s \end{array}\right\} \left.\begin{array}{l} \text{Total } f+s \\ \text{Oxygen } o \end{array}\right\} f+s+o$$

$$\text{Oxide of zinc } n+r-f-s-o$$

$$\begin{array}{lr} \text{Flux added} \quad . \quad . & r \\ \hline \text{Earthy matter} \quad . \quad . & s-r \end{array}$$

The following is an actual experiment by Berthier :—

$$\begin{array}{lr} 100 \text{ crude ore}=\text{calcined ore} \quad . \quad . \quad . \quad . \quad . \quad . & 88\cdot3 \\ 10 \text{ kaolin (china clay) acted on by acids} \quad . \quad . \quad . & 10\cdot0 \\ 7 \text{ marble}=\text{lime} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . & 4\cdot0 \\ \hline & 97\cdot3 \end{array}$$

$$\begin{array}{l}\text{Gave metal} \quad . \quad . \\ \text{Gave slag} \quad . \quad . \end{array} \left.\begin{array}{r} 45\cdot3 \\ 16\cdot3 \end{array}\right\} \begin{array}{l} \text{Total} \quad . \quad . \\ \text{Oxygen} \quad . \end{array} \left.\begin{array}{r} 61\cdot3 \\ 19\cdot4 \end{array}\right\} \text{Total} \quad . \quad . \quad 80\cdot7$$

$$\text{Oxide of zinc} \quad . \quad 16\cdot6$$

$$\begin{array}{lr} \text{Fluxes added} \quad . \quad . \quad . \quad . \quad . \quad . \quad . & 14\cdot0 \\ \text{Earthy matters} \quad . \quad . \quad . \quad . \quad . \quad . \quad . & 2\cdot0 \end{array}$$

The above result was confirmed by humid analysis, showing at once the exactitude of the process.

*Determination of amount of Zinc by the Humid Process, in Ores of the First Class.*—Dissolve 50 grains of the finely pulverised ore in nitric acid, evaporate to dryness, allow to cool. Digest the cold mass with a little dilute nitric acid, gently warming during the digestion, add water, and then filter. To the filtered solution add excess of caustic ammonia, gently warm, and filter. The excess of caustic ammonia dissolves the oxide of zinc which it at first threw down, as well as any oxide of manganese that may be present. This solution containing the zinc, and probably manganese, must be separated from the precipitate produced by the ammonia by filtration, the insoluble matter in the filter washed with water containing a little ammonia, and the washings so obtained added to the first strong filtrate. If no manganese be present, sulphide of ammonium may be now added to the filtered liquid until it produces no further white precipitate of oxide of zinc. The liquid and precipitate must now be allowed to stand in a warm place for about an hour, then filtered, and the sulphide of zinc on the filter washed with water containing a little sulphide of ammonium. After a few washings, it is to be dissolved in dilute hydrochloric acid, and, if necessary, the solution filtered. To the filtered solution is added excess of carbonate of soda: carbonate of zinc is thrown down, which in its turn is collected on a filter, washed, dried, separated from the filter, ignited, and weighed. Four-fifths of its weight is metallic zinc. If by previous experiment by blowpipe, or otherwise, manganese were found to be present, the ammoniacal solution containing the mixed oxides must be thus treated:—Excess of acetic acid is to be added to it, and a stream of sulphuretted hydrogen gas passed through it until no further precipitation takes place; by this means the whole of the zinc is deposited as sulphide, whilst the manganese remains untouched in the liquid. The sulphide of zinc is to be collected on a filter and treated with hydrochloric acid, &c., as just described.

### ASSAY OF ORES OF THE SECOND CLASS.

The silicates of zinc are not reducible by charcoal alone; but when in contact with substances which have the property of combining with silica, they are reduced completely, even at a moderate temperature. All the modes of assay just described for ores of the first class apply to those of the second, with the exception that the flux, instead of being merely reducing, must have a true fluxing property also: lime or magnesia are good fluxes.

*Humid determination of Zinc in Ores of the Second Class.*—Ores of this class are best decomposed by strong hydrochloric acid with a small admixture of nitric acid. When thoroughly decomposed, and the solution evaporated to dryness, it is moistened with hydrochloric acid, and treated exactly as described for Ores of the First Class.

### ASSAY OF ORES OF THE THIRD CLASS.

In order to assay the substances containing sulphur which belong to this class, they must be roasted, and then treated as the ores of the first and second class. Sulphide of zinc may be roasted without difficulty; and when the operation is made with care, the roasted ore contains neither sulphur nor sulphuric acid. The only precaution necessary to observe is, that the heat must be carefully regulated at first, in order to avoid fusion which might take place, especially when a certain amount of sulphide of iron is present. Towards the end the heat may be increased, to decompose any sulphate that may be formed. Both a reducing and fusing substance must be added in this case, as in the last, in order to determine the fusion of the gangue.

*Humid Determination of Zinc in Ores of the Third Class.*—These ores are to be finely pulverised, treated with strong nitric acid, at first with a gentle heat; and lastly, boiled until the sulphur separates in bright yellow transparent globules, as described under the Humid Assay of Copper Ores of the Second Class. The solution so obtained is to be evaporated to dryness, moistened with hydrochloric acid, and treated as described for ores of the first class.

If ores of this class, or of either of the two former, contain copper, they must be thus treated :—

The ore is to be decomposed by an appropriate acid, evaporated to dryness, moistened with hydrochloric acid, water added, and the solution filtered. A current of sulphuretted hydrogen gas is now to be passed through the solution until, even after violent agitation, it smells strongly of it. It is now to be filtered, and the black precipitate on the filter contains all the copper as sulphide of copper, that substance being insoluble in dilute acid, whilst in a solution acidulated with either of the strong mineral acids—as nitric, hydrochloric, or sulphuric—zinc is not at all acted on by sulphuretted hydrogen. The solution, now freed from copper, is placed in an evaporating basin and boiled for about a quarter of an hour: nitric acid is then added to peroxidise all the iron present, and the solution allowed to cool. When cold, the zinc is separated by means of ammonia, and the ammonical solution treated as already described.

### FOURTH CLASS. ALLOYS.

The alloys of zinc with iron, copper, and tin, may be assayed by heating them to whiteness for about an hour in a charcoal crucible with an earthy flux (silicate of lime is the best), and weighing the resulting button: the loss will be nearly equivalent to the quantity of zinc present.

*The Humid Determination of Zinc in Substances of the Fourth Class.*—These substances are treated precisely as described under the heads Humid Determination of Zinc in First, Second, and Third Classes.

### VOLUMETRIC DETERMINATION OF ZINC.

Fresenius * gives the following methods for the volumetric determination of zinc.

Several methods have been proposed for the volumetric determination of zinc. The most suitable method for technical purposes † seems to be that based on the precipitation of an ammoniacal solution with standard sulphide of sodium.

This method was originally proposed by Shaffner: it has been the subject of a variety of modifications. After this method, with its modifications, have been detailed, I shall proceed to describe the method of H. Schwarz, and then that of Carl Mohr. The two first methods require the zinc in ammoniacal solution, while for the last method an acetic acid solution is employed.

#### 1. METHOD OF SCHAFFNER,‡ MODIFIED BY C. KÜNZEL,§ AS EMPLOYED IN THE BELGIAN ZINC-WORKS; DESCRIBED BY C. GROLL.||

#### *a. Solution of the Ore and Preparation of the Ammoniacal Solution.*

Powder and dry the ore.

Take 0·5 grm. in the case of rich ores, 1 grm. in the case of poor ores, transfer to a small flask, dissolve in hydro-

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* 4th English edition, p. 653, published by Churchill and Sons.

† It is very extensively employed in zinc works.

‡ Journ. f. prakt. Chem. 73, 410.

§ Ibid. 88, 486.

|| Zeitschrift f. anal. Chem. 1, 21.

chloric acid with addition of some nitric acid by the aid of heat, expel the excess of acid by evaporation, add some water, and then excess of ammonia. Filter into a beaker, and wash the residue with lukewarm water and ammonia, till sulphide of ammonium ceases to produce a white turbidity in the washings. The oxide of zinc remaining in the hydrated sesquioxide of iron is disregarded. Its quantity, according to Groll, does not exceed 0·3—0·5 per cent. This statement probably has reference only to ores containing relatively little iron; where much iron is present the quantity of zinc left behind in the precipitate may be not inconsiderable. The error thus arising may be greatly diminished by dissolving the slightly washed iron precipitate in hydrochloric acid, and adding excess of ammonia. But the surer mode of proceeding is to add to the original solution—after evaporating off the greater part of the free acid as above, and allowing to cool—dilute carbonate of soda nearly to neutralisation, then to precipitate the sesquioxide of iron with acetate of soda, boiling to filter, and wash. The washings, after being concentrated by evaporation, are added to the filtrate, and the whole is then mixed with ammonia, till the first-formed precipitate is redissolved.

If the ore contains manganese—provided approximate results will suffice—digest the solution of the ore in acids, after the addition of excess of ammonia and water, at a gentle heat, for a long time, and then filter off, with the iron precipitate, the hydrated protosesquioxide of manganese which has separated from the action of the air. The safer course—though undoubtedly less simple—is, after separating the iron with acetate of soda, to precipitate the manganese by passing chlorine, or by adding bromine and heating.

If lead is present, it is separated by evaporating the aqua regia solution with sulphuric acid, taking up the residue with water and filtering; then proceed as directed.*

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* Concerning the direct treatment of roasted zinc ores with a mixture of carbonated and caustic ammonia, comp. E. Schmidt (*Journ. f. prakt. Chem.* 51, 257). By this treatment the oxide of zinc, which was combined with carbonic acid, is dissolved, whilst that combined with silicic acid is for the most part left undissolved.

### *b. Preparation and Standardising of the Sulphide of Sodium Solution.*

The solution of sulphide of sodium is prepared either by dissolving crystallised sulphide of sodium in water (about 100 grm. to 1,000—1,200 water), or by supersaturating a solution of soda, free from carbonic acid, with sulphuretted hydrogen, and subsequently heating the solution in a flask to expel the excess of sulphuretted hydrogen. Whichever way it is prepared, the solution is afterwards diluted, so that 1 c. c. may precipitate about 0·01 grm. zinc. Prepare a solution of zinc, by dissolving 10 grm. chemically pure zinc in hydrochloric acid, or 44·122 grm. dry crystallised sulphate of potash of zinc in water, or 68·133 grm. dry crystallised sulphate of potash and zinc in water, and making the solution in either case up to 1 litre with water.

Each c. c. of this solution corresponds to 0·01 grm. zinc. Now measure off 30—50 c. c. of this zinc solution into a beaker, add ammonia till the precipitate is redissolved, and then 400—500 c. c. distilled water. Run in sulphide of sodium as long as a distinct precipitate continues to be formed, then stir briskly, remove a drop of the fluid on the end of a rod to a porcelain plate, spread it out so that it may cover a somewhat large surface, and place in the middle a drop of pure dilute solution of chloride of nickel. If the edge of the drop of nickel solution remains blue or green, proceed with the addition of sulphide of sodium, testing from time to time, till at last a blackish grey coloration appears surrounding the nickel solution. The reaction is now completed, the whole of the zinc is precipitated, and a slight excess of sulphide of sodium has been added. The precise depth of colour of the nickel must be observed and remembered, as it will have to serve as the stopping signal in future experiments. To make sure that the zinc is really quite precipitated, you may add a few tenths of a c. c. more of the reagent, and test again; of course the colour of the nickel drop must be darker. Note the number of c. c. used, and repeat the experiment, running in at once the necessary quantity of the reagent *less* 1 c. c., and then adding 0·2 c. c.

at a time, till the end reaction is reached. The last experiment is considered the more correct one. The sulphide of sodium solution must be restandardised before each new series of analyses—that is, if it is kept in bottles containing air; if, on the contrary, oxygen is excluded by passing the air through an alkaline solution of pyrogallic acid previous to its entering the bottle, the solution would without doubt keep unaltered.

### *c. Determination of the Zinc in the Solution of the Ore.*

Proceed in the same way with the ammoniacal solution prepared in *a* as with the known zinc solution in *b*. Here also repeat the experiment, the second time running in at once the required number of c. c., less 1, of sulphide of sodium, and then adding 0·2 c. c. at a time, till the end-reaction makes its appearance. The second result is considered the true one. There are three different ways in which this repetition of the experiment may be made. You may either weigh out at the first two portions of the zinc ore, or you may weigh out double the quantity required for one experiment, make the ammoniacal solution up to 1 litre, and employ ½ litre for each experiment, or lastly, having reached the end-reaction in the first experiment, you may add 1 c. c. of the known zinc solution, which will destroy the excess of sulphide of sodium, and then run in sulphide of sodium in portions of 0·2 c. c. till the end-reaction is again attained. Of course, in this last process to obtain the second result, you deduct from the whole quantity of sulphide of sodium used the amount of the same, corresponding to 1 c. c. of the zinc solution.

If the ore contains copper, which frequently occurs in the case of blendes, determine by a preliminary experiment the number of c. c. of sulphide of sodium which are necessary to precipitate the copper, and at the completion of the zinc-analysis deduct them. In this case, let the drop to be tested with nickel solution pass through a small filter on its way to the porcelain plate, in order to avoid the injurious influence of the sulphide of copper on the nickel reaction. If, however, the copper amounts to more than 2 per cent., remove it from the acid solution by sulphuretted hydrogen, evaporate the filtrate with nitric acid, dilute, treat with ammonia, and determine the zinc as above.

In careful hands the error will, according to C. Künzel, never exceed ½ per cent.

### *d. Further Modification of the Process.*

To ascertain the point when the whole of the zinc is precipitated and the sulphide of sodium begins to predominate, Schaffner[*] employed flocks of hydrated sesquioxide of iron, which he produced by the addition of a few drops of sesquichloride of iron to the ammoniacal zinc solution, and which settled at the bottom; while Barreswil[†] used small pieces of white porcelain, which were covered with sesquichloride of iron, and thrown into the ammoniacal zinc solution. Sulphide of sodium is added till the flocks or the pieces of porcelain turn black. In neither case is the end-reaction so exact as with nickel solution.

With the help of lead-paper, however, the point may be hit with great precision. Moisten a piece of white filter-paper with solution of acetate of lead, place it on a layer of blotting-paper, drop some carbonate of ammonia upon it, so as to form a thin coating of carbonate of lead, let the blotting-paper absorb the excess of moisture, and then spread the lead-paper on a porcelain plate. As soon as you imagine the zinc to be nearly all precipitated, lay a small piece of filter-paper on the lead-paper, and then dip the end of a blunt glass rod in the fluid, and press it somewhat gently on the small piece of filter-paper. When the sulphide of sodium begins to be in excess, a brown spot forms on the lead-paper. This lead-paper appears to be more sensitive than the nitroprusside of sodium-paper proposed by Carl Mohr,[‡] which, however, is very serviceable. Fr. Mohr[§] applies the lead-reaction in another manner. He makes an alkaline solution of lead by warming together acetate of lead,

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[*] Journ. f. prakt. Chem. 73, 410.

[†] Journ. de pharm. 1857, 431; Polyt. Centralbl. 1858, 285.

[‡] Dingler's Polyt. Journ. 148, 115.

[§] Mohr's Lehrbuch der Titrirmethode, 2 Aufl. 377.

Rochelle salt and solution of soda; he first places a drop of this on filter-paper, and then close by a drop of the precipitated zinc solution, so that the circle formed by the spreading of the solution to be tested may cut the circle of the lead solution. As soon as the sulphide of sodium begins to predominate, the portion of the circumference of the lead circle, which lies in the other circle, turns black.

### 2. H. SCHWARZ'S METHOD.*

Prepare an ammoniacal solution as in 1, *a*.

Heat gently, and mix with a moderate excess of sulphide of ammonium. Allow the precipitated sulphide of zinc to subside, then filter, using a tolerably large plaited filter of rapidly filtering paper, moistened with boiling water, and warming the fluid to accelerate the operation, which would otherwise require considerable time. Wash the precipitate with warm water mixed with a little ammonia, until the last drops no longer blacken a solution of oxide of lead in soda.

Transfer the filter with the precipitate to a beaker, add a dilute solution of slightly acidified sesquichloride of iron, cover with a close-fitting glass plate, and let the mixture stand for ten minutes; then heat gently. Under these circumstances the sulphide of zinc decomposes completely with the sesquichloride of iron to chloride of zinc, protochloride of iron, and sulphur: $Fe_2Cl_3 + Zn\ S = Zn\ Cl + S + 2FeCl$.

Now add sulphuric acid, and heat gently until the sulphur has agglutinated. Filter, wash the filter, and determine the iron in the fluid as protochloride by permanganate,† 2 eq. iron correspond to 1 eq. zinc. If the quantity of sulphide of zinc is not very great, the filter may be broken, and the sulphide of zinc washed into a flask which already contains the solution of sesquichloride of iron. The great objection to this method lies in the washing of the sulphide of zinc, which, as is well known, is a long and troublesome operation. A possible loss of sulphuretted hydrogen on mixing the sulphide of zinc with sesquichloride of iron may be prevented by conducting the decomposition in a flask, connected with a U-tube containing sesquichloride of iron.

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* See his Anleitung zu Maassanalysen, Nachträge, p. 29 (Brunswick). Compare also v. Gellhorn (Chem. Centralbl. 1853, 291), who has made many analyses by Schwarz's method.

† Without doubt the sesquichloride of iron might be replaced by the sesquisulphate, by which means the presence of hydrochloric acid would be avoided.

### 3. Carl Mohr's Method.*

This method is based upon the following considerations :—

I. If a solution of acetate of zinc, acidified with acetic acid, is mixed with an excess of ferricyanide of potassium, the whole of the zinc is thrown down in the form of a reddish yellow precipitate of ferricyanide of zinc $Zn_3\,(Cy_6Fe_2)$.

II. If solution of iodide of potassium is now added in excess, we have this decomposition :—$2[Zn_2\,(Cy_6Fe_2)] + 2KI + 2(\overline{A}, HO) = 3[Zn_2\,(Cy_3Fe)] + 2(KO,\overline{A}) + H_2(Cy_3Fe) + 2I$.

III. 1 eq. liberated iodine corresponds, accordingly, to 3 eq. zinc.

IV. If iodide of potassium is made to act upon ferricyanide of zinc in a neutral fluid the liberated iodine acts upon the ferrocyanide of potassium present in that case, which leads to the formation of a little ferricyanide of potassium ; the remaining free iodine, therefore, will not indicate, with accuracy, the quantity of zinc present. But whereas the reaction actually takes place in acid solution of acetate of zinc, as above directed, it may be assumed that acetate of potassa and free hydroferrocyanic acid are formed ; and as iodine exercises no appreciable action upon the latter substance, the iodine liberated in the process indicates, with tolerable accuracy, the amount of zinc present.

The process is as follows :—

Treat the ore with aqua regia, as in 1, *a*, and drive off the greater part of the free acid ; nearly neutralise with carbonate of soda, add acetate of soda in excess, boil, filter, and wash with boiling water mixed with a little acetate of soda. The solution is iron-free ; it contains the whole of the zinc, but, in presence of manganese, also the whole of the latter metal. Hence the process is not applicable in the presence of manganese.

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* Dingler's Polyt. Journ. 148, 115.

Mix the solution of zinc, prepared as directed, with ferricyanide of potassium in slight excess, i.e. until a sample of the clear supernatant fluid gives a blue precipitate with a salt of protoxide of iron. Then add a sufficient quantity of iodide of potassium. The fluid acquires a brown colour, in consequence of the liberation of iodine; the white precipitate of ferrocyanide of zinc is suspended in the brown fluid.

Determine now the free iodine by means of hyposulphite of soda, and calculate 3 eq. zinc for each eq. iodine. The results obtained by C. Mohr are very satisfactory. The method can be employed only if the acetic acid solution contains no other heavy metal besides zinc, and, more particularly, no manganese.

## BLOWPIPE REACTIONS OF ZINC.

**Zinc Blende, Black Jack, Sulphide of Zinc.**—*Alone*, decrepitates violently. Suffers no remarkable change on ignition; does not fuse, and gives off but a very slight odour of sulphurous acid, being very difficult to roast.

*On charcoal*, an annular deposit of oxide of zinc is formed when heated violently in the outer flame.

*Soda* attacks it feebly; but the zinc is reduced in a good fire, with the deposition of oxide of zinc on the charcoal.

**Carbonate of Zinc, Calamine.**—*Alone*, gives off no water, but becomes a white enamel, which behaves like oxide of zinc.

**Oxide of Zinc.**—*Alone*, becomes deep yellow when heated. This assay must be made by daylight. It reassumes its white colour on cooling. It does not fuse, but gives off a vivid light during incandescence. It is gradually evaporated in the reducing flame, during the continuance of which a yellow ring is deposited on the charcoal, which becomes white on cooling.

*With borax* it fuses readily, and gives a transparent glass, which, with a large proportion of oxide, becomes milky by flaming. It assumes an enamel-white appearance on cooling. In the reducing flame the metal sublimes, and covers the charcoal with a white film.

*With microcosmic salt* it behaves as with borax, except that the metal sublimes less readily with the first than the second.

*Soda* does not dissolve it; but acted on by this reagent on charcoal, it is reduced, and covers the charcoal with a coating of oxide.

# CHAPTER XIV.

## ASSAY OF MERCURY.

**Mercury** is found in the native or metallic state, and as sulphide or cinnabar:—

Native mercury, Hg.
Sulphide of mercury, cinnabar, $Hg^2S$.
Bituminous sulphide of mercury.

There are other minerals of mercury met with, but hitherto not in sufficient quantity to be worked for the metal. They are:—

Zinciferous subsulphide of mercury.
Zinciferous sulphide of mercury.
Selenide of mercury.
Subchloride of mercury.
Iodide of mercury.
Silver amalgam (*see* Silver).

*Assay of Mercurial Ores.*—The determination of mercury is always made by distillation. In case the mercury is present in the form of native mercury, or oxide of mercury, it is distilled without any addition. The ore (say from 500 to 1,000 grains) is placed in an iron or earthenware retort, which is set over a suitable fire, and the heat raised gradually, and kept up, until the whole of the mercury has passed over. The mercury which passes over is collected either in the neck of the retort, or a receiver fitted for that purpose—such as a glass flask kept cool by affusion with water. When but a small quantity is operated upon (say 150 to 200 grains), it is most convenient to use a glass retort, or bent tube retort, heating it gradually over a charcoal fire, taking care to keep the upper part so hot, that no metallic mercury may adhere to it. It must be heated nearly to the melting point of the glass, and until all the mercury has come over.

When the operation is finished, the neck is cut off, weighed, the mercury detached, and weighed again: the loss of weight is the amount of mercury. Or the metal may be detached by means of a feather, and allowed to fall into a basin of water, which, if heated for a few seconds, will cause the mercury to collect into one globule: the water may be decanted, and the mercury dried at the ordinary temperature, and weighed.

The mercury wholly condenses in the neck of the retort, under the form of a metallic dew. Some may by chance pass off; but in order to prevent such an occurrence, the beak of the retort is plunged into water, or a small dossil of linen, moistened with water, introduced into the neck, the end of which is plunged into water, by which means the neck of the retort is kept constantly cool, and the mercury is found deposited on the linen, from which it may be detached by shaking in water.

When large quantities of substances containing mercury are operated upon, it is necessary to heat very strongly towards the end, in order that the centre of the mass may receive a sufficient amount of heat to effect its decomposition. Naked glass retorts cannot be used; and either coated glass or porcelain retorts must be employed. In the large way, as in the distillation of amalgams, &c., cast-iron retorts are used.

As before stated, all substances containing mercury, either in its metallic state or as oxide, are distilled without addition, but with the others it is necessary to employ some reagent, which will separate and retain the sulphur, selenium, &c.; which reagent may be a metal, as iron, copper, or tin; or black flux, or a mixture of quick-lime and charcoal: iron filings are most often used. For cinnabar about 50 per cent. of iron filings is required, in order to prevent any of it being sublimed; the true quantity required is only about 24 per cent., but an excess is necessary, in order, as before stated, to prevent loss: 50 per cent. of iron filings may be employed for the selenides, &c. When black flux is used, from about 50 to 70 per cent. is employed. Caustic lime may be employed in the proportion of 30 per cent. mixed with 30 per cent. its weight of charcoal. After the ore to be assayed is carefully mixed with any of the above fluxes, it is always advisable to cover it, when in the retort, with a thin layer of the flux employed, in order to avoid all chance of any loss.

Berthier, who experimented with an ore containing arsenic, realgar, &c., and cinnabar from Huanca-Vélica, in Peru, found, after manifoldly varied fruitless experiments, the following method best adapted to its examination for mercury:—

The ore was heated in a retort with four to five times its weight of litharge. From the litharge, the sulphide of arsenic, &c., a fusible slaggy mass was formed while the cinnabar was decomposed into sulphurous acid and metallic mercury. The mercury volatilised completely at a moderate heat, and collected in the fore part of the neck of the retort and in the receiver. The single precaution which must be observed for the success of the assay, consists in only gradually and moderately heating the clay or glass retort, in order to prevent its being perforated by the corroding effect of the litharge before the operation is ended.

If the assay sample is extremely poor in mercury, the ordinary assay method becomes somewhat inconvenient and uncertain, on account of the large quantity which must then be subjected to distillation in the assay. For this case Berthier found it more appropriate to digest the assay sample with aqua regia, wash it thoroughly, evaporate the whole mass of fluid to dryness, and then treat the dry mass, which contains all the mercury as chloride, further in the dry way. He found that if chloride of mercury (corrosive sublimate) is heated with litharge, it volatilises without undergoing any change. If, besides the litharge, coal-dust is also added, or if instead of it metallic lead is used in great excess, the chloride is reduced to subchloride, which volatilises, but not the smallest drop of mercury is thus produced. The best reducing agent for the chloride of mercury contained in the dry mass, is black flux, of which three parts by weight are used. Since the mass to be subjected to distillation has been greatly diminished by the treatment with aqua regia, and the subsequent evaporation, and no high heat is now required for the decomposition, the distillation may be performed in a glass retort. When the gangue in the poor ore is carbonate of lime, all the lime is dissolved out, *before* the treatment with aqua regia, by moderately strong acetic acid.

By this method the smallest trace of mercury in an ore or amalgamation product can be shown and determined by its weight.

*Assay for the Amount of Cinnabar in an Ore.*—The ore to be assayed is distilled, without addition, in a glass retort, and the sublimed cinnabar collected and weighed. The ores containing mercury combined with sulphur are often mixed with bituminous matters and carbonate of lime: then, when an assay is to be made for cinnabar, it often happens that a portion of it is decomposed, either by the carbon present, or by the aid of the bituminous matter and lime, and a little metallic mercury is driven off with the cinnabar. In this case, having weighed the mixture of cinnabar and mercury, the mixture is treated by nitric acid, which dissolves only the latter, and pure cinnabar remains, whose weight is taken, and the quantity of mercury dissolved ascertained by the difference; and from that the quantity of cinnabar calculated which that quantity of mercury would yield. Every 86 parts of mercury furnish about 100 of cinnabar.

If the gangue of the ore be fixed in the fire, the assay may be made by mere calcination, and the loss of weight will correspond either to the metallic mercury, oxide, or sulphide it may contain.

## VOLUMETRIC ESTIMATION OF MERCURY.

The process we have found most trustworthy is that of M. J. Personne, described in the 'Comptes Rendus,' lvi. 63, as follows. The author says:—

'Two methods have hitherto been used for the estimation of mercury, the wet and the dry way—one for solid compounds, the other for mercurial solutions. In the wet way it is estimated in the state of protochloride, or, better still, as metallic mercury by means of appropriate reducers, or as a sulphide. This method, which necessarily occupies a long time, is not always so exact as might be desired. The dry way, though more easily executed, and giving more exact results, still requires a considerable length of time. It applies but indirectly to the estimation of a mercurial solution, which renders it almost useless in this instance. Being under the necessity of estimating a number of mercurial solutions by a certain time, I was compelled to find some more certain and rapid means of estimation than either of those I have enumerated. The following is the result of my investigations.

'The process at which I have arrived, after many fruitless attempts, is founded on a well-known fact—that a combination of iodide of mercury with iodide of potassium, forming the double iodide of Polydore Boulay, HgI,KI, gives a colourless solution. Thus, two solutions in equal quantities, one containing one equivalent of bichloride of mercury, the other two equivalents of iodide of potassium, being mixed, by pouring the mercurial solution into that of the iodide of potassium, iodide of mercury will be produced by the contact of the two solutions, which dissolves in proportion to its formation, until the mercurial solution added is equal in volume to that of the alkaline iodide used, according to the following equation:—

$$HgCl+2KI=HgI,KI+KCl.$$

The slightest excess of bichloride causes the formation of a persistent red precipitate, giving the liquid a very perceptible red tint even by artificial light. This coloration, which indicates that the saturation is complete, gives to this mode of estimation a precision and nicety quite as great as that of litmus used to ascertain the saturation of an acid by a base. The mercurial solution must always be poured into the alkaline iodide, not the alkaline iodide into the mercurial solution; otherwise, though the last reaction may be the same, it is impossible to obtain exact results, because the iodide of mercury produced, not being brought simultaneously with its formation (in a nascent state) into contact with the alkaline iodide with which it is to combine, becomes sufficiently cohesive to dissolve but slowly in the iodide of potassium. Thus, in operating with the same liquids, the quantity of alkaline iodide which must be added to dissolve the iodide of mercury precipitated varies according to the time employed in effecting the estimation, and that in considerable proportions. I have no doubt that it is through operating in this way that iodide of potassium has hitherto been rejected as a medium for the exact estimation of mercury.

'Two normal liquids are necessary to effect this estimation.

'1. *Normal Standard Solution of Iodide of Potassium.*—Obtained by dissolving 33·20 gr. of pure iodide of potassium in water enough to make 1 litre of solution. 10 cubic centimetres of this solution represent 0·1 of metallic mercury.

'2. *Normal Standard Solution of Bichloride of Mercury.*—Prepared by dissolving 13·55 gr. of bichloride of mercury in water so as to make 1 litre of solution. The solution of mercurial salt is facilitated by the addition of 5 equivalents, or 30 grammes, of chloride of sodium, which has no influence on the reaction, like all neutral alkaline salts; 10 cubic centimetres of this solution also represent 0·1 of mercury. Of these 10 centimetres, divided into 100 parts, each division represents 0·001 of mercury. This mercurial solution serves to test the purity of the alkaline iodide solution or to give the standard of an unknown solution.

'Liquids ten times more diluted may be prepared without injuring the nicety of the reaction or the exactness of the results; fractions of a milligramme may thus be estimated.

'The estimation is effected in the following manner:—10 cubic centimetres of a normal solution of iodide being measured into a small saturating vessel, pour into it, constantly shaking the vessel, the solution of bichloride, measured in Gay-Lussac's burette. If the two liquids are pure, it will require exactly 100 divisions of the burette before the light red tint appears in the saturated liquid to indicate the close of the operation. When the mercurial solution is weak a proportionally larger quantity must be added; if, on the other hand, it is too strong, less must be added. As will be perceived, this is very similar to the chlorometric process.

'This new method of estimating mercury being applicable only to bichloride, it became desirable to extend its application to a greater number of mercurial compounds, if not to all. This side of the question presented difficulties not easily resolved in a satisfactory manner. It was, in fact, necessary to transform all the mercurial compounds into a perfectly neutral solution of bichloride. I was obliged to set aside successively the use of aqua regia, and even of hypochlorous acid. The great volatility of bichloride of mercury, even in a boiling solution, caused too great a loss. M. Rivot's process—that is to say, the action of chlorine in a solution of hydrate of potash or soda—is perfectly successful. Take, for instance, the estimation of mercury in cinnabar. Reduce one gramme of cinnabar to a fine powder. Weigh it on paper, and introduce it into a matrass. Pour into the matrass 20 cubic centimetres of a caustic soda solution, with which mix the paper and its contents by quickly shaking; then send a current of chlorine, which need not be washed, into the liquid. The action of the chlorine produces a slight heat, which is gradually brought to boiling-point, by which time all the matter will have disappeared. To ensure success, the temperature must be carefully managed at the commencement. If it is raised too quickly, part of the matter remains undissolved. The solution being complete and saturated with chlorine, it is kept boiling long enough to expel all the excess of chlorine. The boiling may be prolonged without incurring any loss of bichloride, which is not volatile in presence of alkaline chloride. The solution when cooled is poured into a graduated tube. The matrass as well as the tube for conducting the chlorine are washed two or three times with water, and the washing added to the original liquid, so as to form 100 cubic centimetres of solution. I effected the estimation with the standard solution of iodide, of which 10 centimetres represent 1·0 of mercury. To saturate these 10 centimetres it required 115 divisions of the chloromercurial solution. These 115 divisions contain then 0·10 of mercury. Now, as all the mercury contained in the analysed cinnabar is spread through the 10,000 divisions of solution, we have the quantity of mercury found by the experiment by means of a simple proportion.'

## BLOWPIPE REACTIONS OF MERCURY.

**Mercury.**—The compounds of mercury are all volatile, and cannot, in consequence, be distinguished by their reaction with any of the fluxes. Substances containing mercury are assayed by being mixed with a little tin, iron filings, or oxide of lead, and heating the mixture to redness in the closed tube or matrass. In this operation the mercury is reduced, and collects in the coldest part of the tube as a greyish powder, which being brought together by the end of a feather, collects as metallic globules. When the quantity is very small, the globules may be distinguished by aid of the microscope.

## ORES OF MERCURY.

**Cinnabar, Sulphide of Mercury.**—*Alone*, on charcoal, it volatilises without residue, giving off an odour of sulphurous acid. In the matrass it sublimes, giving a blackish sublimate. In the open tube, it gives, by roasting, mercury and sublimed cinnabar. In the matrass, with soda, globules of mercury are obtained.

**Chloride of Mercury, Horn Mercury.**—On charcoal, volatilises without residue. In the matrass, gives a white sublimate. With soda, in the matrass, gives much mercury in globules.

*With microcosmic salt*, fused on the brass wire, it communicates a fine azure blue colour to the flame, indicative of chlorine.

# CHAPTER XV.

## ASSAY OF SILVER.

All argentiferous substances may be divided into two classes, as follows :—

Class I.—All minerals containing silver,

Silver glance ($AgS$) containing 87 per cent. of Ag.
Brittle silver ore ($6AgS,SbS_3$) containing 70,4 per cent. of Ag.
Light red silver ore ($3AgS,AsS_3$) containing 65,4 per cent. of Ag.
Dark red silver ore ($3AgS,SbS_3$) containing 59 per cent. of Ag.
Light and dark fahlerz (argentiferous grey copper ore), containing from 5,7 to 18-31·8 per cent. of Ag.
Argentiferous sulphide of copper ($Cu_2S,AgS$) containing 53 per cent. of Ag.
Polybasite ($9(Cu_2S,AgS)+(SbS_3,AsS_3)$) containing 72-94 per cent. of Ag.
Slags.
Cupel bottoms.
Dross.
Litharge, etc.

Class II.—Metallic silver and alloys, either native or otherwise.

### *General Observations on the Assay of Ores and Substances of Class No. 1.*

In order to separate silver from this class of substances, an alloy of the precious metal with lead must be formed. The different methods by which this object can be obtained are the following : firstly, fusion with a reducing flux ; secondly, fusion with oxidising reagents ; thirdly, scorification.

All substances containing lead in the state of oxide, such as carbonates, phosphates, &c., are fused directly with a reducing flux, as also are slags, old cupels, litharge, &c. All plumbiferous sulphides, &c., containing silver, are assayed as for lead by the processes already pointed out, taking care to follow the method which gives the largest proportion of lead.

All argentiferous minerals containing copper may be assayed as copper ores; because an alloy of copper and silver can be cupelled by means of lead.

In making assays of silver with lead or copper, it is sometimes necessary to commence the operation by roasting the ore; under other circumstances, also, argentiferous matters are roasted.

There is nothing very particular to be observed in this roasting; the temperature alone requires attention by managing well at the commencement of the operation, in order to avoid softening, and especially to avoid a very rapid disengagement of arsenical vapours, because a very considerable amount of silver may be lost by that means.

All substances which contain reducible oxides are fused with a reducing flux, as also those from which charcoal separates metals which alloy with lead, or metals which do not hinder the process of cupellation; but it is necessary to add to the reducing flux a certain proportion of litharge, in order to produce metallic lead, with which the silver may alloy. A mixture of metallic lead and any suitable flux may be substituted for that of litharge and a reducing flux; but the latter is preferable, because the lead produced is uniformly diffused throughout the whole mass of flux, &c., not allowing a particle of silver to escape its action.

The reducing agent employed in nearly all assays is charcoal either in its ordinary state, or as it is found in black flux. Starch and other analogous substances may be, as before mentioned, substituted for it: crude argol is, however, the best reducing agent. The portion employed must be varied according to circumstances, so that the silver-lead produced be not too rich, or that too great a proportion of lead be reduced. If the silver-lead be too rich, much of the precious metal may be lost in the slag, and if too great a quantity of lead be produced, silver is again lost, owing to the long exposure to the fire during cupellation; and indeed this is the most fruitful cause of loss, for more is lost in this manner than by having too little lead produced. In order to know the right proportions, the following data will serve as a guide:—1 part of charcoal reduces about 30 parts of lead from litharge, and 1 part of black flux reduces about 1 part of lead.

The fluxes employed in this kind of assay are litharge, black flux, carbonate of potash or soda, and borax. Litharge is an exceedingly convenient flux, because it occupies very little room, and fuses without bubbling, producing very liquid scoriæ with nearly every substance. Experiment has shown that nearly all argillaceous, stony, and ferruginous substances fuse very well with from 8 to 12 or more parts of litharge. If from $\frac{1}{2}$ to 1 part of black flux, or $\frac{1}{30}$th to $\frac{1}{25}$th of charcoal, be added to 1 of ore, from $\frac{1}{2}$ a part to 1 part of silver-lead will be produced.

Black flux is employed in the fusion of all substances containing a large proportion of alumina, or in which lime is the predominant substance: from 2 to 3 parts of this flux generally suffice: 1 part of litharge is added to the assay, which is wholly reduced, producing nothing but lead.

The carbonates of potash or soda produce exactly the same effects as the alkali of the black flux. A certain quantity of charcoal must, in this case, be added to the assay.

Schlutter fuses the poor refuse of goldsmith's workshops, mixtures of fragments of crucibles, glass, &c., with 2 parts of carbonate of potash, when they are very earthy, and with 1 part only, when they contain much glass, adding, at the same time, to the mixture, a little litharge and granulated lead.

Borax has, like litharge, the advantage of being an universal flux; it is useful especially for the fusion of substances containing much lime; but it is necessary to take great care in the assay, in order to avoid the loss which its boiling up might occasion. This only applies, however, to its use in its ordinary state; if previously fused, that is, used as glass of borax, no particular care need be taken.

## FUSION WITH OXIDISING REAGENTS.

*Litharge.*—The oxidising agents employed in the assay of argentiferous substances are litharge and nitre. Litharge attacks all the sulphides, arsenio-sulphides, &c., and oxidises nearly all the elements, excepting silver, when employed in sufficient quantity, and a quantity of lead equivalent to the oxidisable matters present is reduced, so that there results from the assay a slag containing an excess of oxide of lead, and an alloy of lead and silver, very little contaminated with foreign metals, if no copper be present, and which can be submitted directly to cupellation. This method of assay is exceedingly convenient and quick.

The pulverised mineral is well mixed with litharge, and the mixture placed in a crucible, which may be very nearly filled, as there is scarcely any boiling up when the pot and its contents are submitted to the fire. A thin layer of pure litharge is placed above the mixture, the whole is then heated rapidly, and as soon as the litharge, &c., is completely fused, the crucible is taken from the fire. It is inconvenient to heat it for any length of time, on account of the corrosive action litharge has on the substance of the crucible, which it rapidly destroys.

The proportion of litharge which must be employed depends upon the nature and quantity of oxidisable matters present in the ore. It ought in general to be very great, because it is absolutely necessary that no sulphurous matters be present, so that the slag may not contain the least trace of silver. But it is known how much litharge is required to decompose the metallic sulphides. Pyrites requires about 50 parts; mispickel, blende, sulphide of antimony, copper pyrites, grey cobalt, and grey copper, require from about twenty-five to about forty times their weight. For sulphide of bismuth 10 are sufficient, and for galena or sulphide of silver but 4 or 5 parts need be employed. The proportion of litharge will not be so great for a mineral containing much stony gangue as for one entirely metallic. Experiment has proved, that the assay of rough schlichs, such as those treated in the large way by amalgamation, can be made very exactly with from 10 to 12 parts of litharge.

Alloys of silver with the very oxidisable metals can be assayed by means of litharge, such as those of iron, antimony, tin, zinc, &c.; but in order to have a successful result the alloys should be reduced to a very fine state of division, so that they must be at least granulated; and it is very often necessary to repeat the operation several times on the fresh alloy of lead produced.

The method of assay just pointed out is inconvenient, on account of the large quantity of lead it produces; pyrites giving 8½ parts, copper pyrites and blende 7 parts, sulphide of antimony and grey copper about 6 parts, &c. In order to avoid this inconvenience, part of the oxidation can be performed by means of nitre. Nitre alone, employed in excess, oxidises all metallic and combustible substances found with silver, and even, under certain circumstances, a portion of the silver itself; but when the proportion is insufficient to oxidise the whole, and when the mixture contains at the same time litharge, after the nitre has produced its action, the litharge acts in its turn on the remainder of the oxidisable substances, and the resulting lead carries down the silver set free. So that, by employing suitable proportions of nitre and litharge, all the silver contained in oxidisable minerals may be extracted, and any quantity of lead required may be thus alloyed with it.

As to the requisite proportion of nitre, it can be come at by practice, aided by the following data. It requires about 2½ parts of nitre to completely oxidise iron pyrites, 1½ for sulphide of antimony, and ⅗ for galena.

This determination can be ascertained at once as follows: fuse 1 part of the mineral with 30 of litharge, and weigh the resulting button of lead; and having fixed upon the quantity of lead necessary to carry on the cupellation properly, deduct it from the whole weight of the button, and the difference will be the amount of lead necessary to leave the slag in the state of oxide; and as it has been proved by experiment that 1 part of lead requires ·25 to ·30 of nitre, that is, from 25 to 30 per cent., it is easy to calculate the quantity necessary to be added.

When the ore contains sulphur, the latter forms with the nitre sulphate of potash, which swims on the slag without combining with it.

The assay of silver ores by means of nitre is advantageous and useful in a variety of cases. If we wish to determine, for example, very exactly the percentage of silver in a poor galena, a large quantity, say $\frac{1}{4}$ of a pound, must be fused with about an ounce or an ounce and a half of nitre, and a quarter of a pound of carbonate of soda, or better still, the same quantity of litharge, one of either of which must be employed to flux the gangue and temper the deflagration. After the fusion, all the contained silver will be found alloyed with a very small quantity of lead.

Sometimes the assay is made with a larger quantity of nitre than is requisite for the oxidation, and when the mixture is perfectly fused a certain quantity of metallic lead is added, taking care to cover the whole surface of the mixture, either by using granulated lead or a convenient mixture of litharge and charcoal, or litharge and galena. The shower of metallic lead passing through the fluid mass alloys with all the silver it finds in its passage, and so concentrates it.

This process, however, cannot always be confidently employed. If an excess of nitre be employed with substances susceptible of forming peroxides capable of attacking silver, such as some cupreous substances, the lead added reduces the greater part, but not the whole of the silver in the ore, so that the assay will not be perfect.

### *Special Directions for the Crucible Assay of Ores and Substances of the First Class.*

The ores and substances belonging to this class may, for the convenience of assay, be further subdivided on the following principle. It has already been seen that sulphur, and other substances having a great affinity for oxygen, reduce metallic lead from litharge in proportion to the amount of reducing matter present; and as it is necessary in this kind of assay that no more than a certain quantity of lead alloy should be submitted to cupellation, some kind of control must be exercised by the assayer, to keep the quantity of lead reduced in due and proper bounds. This is readily accomplished by what is called a 'preliminary assay,' by which all ores and substances of this class are divided into three sections:—1stly, ores which, on fusion with excess of litharge, give no metallic lead, or less than their own weight; 2ndly, those which give their own weight, or nearly their own weight, of metallic lead; 3rdly, those which give more than their own weight of metallic lead. The preliminary or classification assay is thus conducted:— 1.295-grams.

Carefully mix 20 grains of the finely pulverised ore (all silver ores must be passed through a sieve with 80 meshes to the linear inch) with 500 grains of litharge; place the 32,375 mixture in a crucible which it only half fills; set the crucible, after careful warming, in a perfectly bright fire, and get up the heat as rapidly as possible, so as to finish the operation in a short time, to prevent the action of the reducing gases of the furnace on the oxide of lead, because, if a great length of time were taken in the operation, a portion of the lead reduced might be traceable to the furnace gases, and the result of the experiment vitiated. After the contents of the crucible are fully fused, and the surface perfectly smooth, the crucible may be removed and allowed to cool, and when cold broken. One of three circumstances may now present itself to the assayer: 1stly, no lead, or less than 20 grains, has been reduced; 2ndly, 20 or nearly 20 grains, more or less, may be reduced; and 3rdly, more than 20 grains may have been reduced. 12,950

Now, as it has been already stated, 200 grains of lead alloy is a suitable amount to cupel; and as 200 grains is the best quantity of ore to submit to assay, it will be evident that ores and substances of the second section, or those bodies which give their own weight, or nearly their own weight, of lead alloy, simply require fusion with a suitable quantity of litharge and an appropriate flux. Ores of the first section require the addition of a reducing agent, in quantity equivalent to the standard amount of lead alloy (200 grains); and ores of the third section require an equivalent quantity of an oxidising agent, or an amount of some body which will oxidise the lead in excess of 200 grains of alloy.

The reducing agent employed is argol; the oxidising agent, nitrate of potash. It is necessary, before commencing an assay of a silver ore, to determine how much lead a given weight of the argol the assayer has in use will reduce, as also how much lead a given weight of nitrate of potash will oxidise. These assays are thus made:—

*Assay of Reducing Power of Argol.*—Carefully mix 20 grains of the argol to be tested with 500 grains of litharge and 200 grains of carbonate of soda; place the mixture in a suitable crucible, and cover with 200 grains of common salt. (It is best to mix two such quantities, and take the mean of the results.) Fuse with the precautions pointed out in assay of substances of the first class, containing lead.

Weigh the resulting buttons, and take a note of the mean weight, which will represent the amount of lead reducible by 20 grains of argol.

*Assay of Oxidising Power of Nitrate of Potash.*—Mix 20 grains of finely powdered nitrate of potash, 50 grains of argol, 500 grains of litharge, and 200 grains of carbonate of soda; cover with 200 grains of common salt, and fuse as above. Weigh the resulting button. Now calculate the amount of lead which should have been reduced by 50 grains of argol, and the difference between that and the amount of lead reduced in this experiment will represent the amount of lead oxidised by 20 grains of nitrate of potash.

Thirty to 32 grains of ordinary red argol reduce about 200 grains of lead; and 23 grains of pure nitrate of potash oxidise about 100 grains of lead. The assayer must, however, adopt the numbers found by himself by experiment, as the samples of argol and nitre may be more or less impure. He must also examine every fresh supply of litharge for the amount of silver it contains, in the following manner:—

*Assay of Litharge for Silver.*—Mix 1,000 grains of litharge with 30 grains (or any other quantity that may be, by experiment, found requisite) of argol, 200 grains of carbonate of soda, and cover with salt, as already directed. Fuse the mixture in a suitable crucible; allow it to cool; break and cupel the button obtained, as hereafter to be described: take a note of the amount of silver obtained; and as 1,000 grains of litharge is the standard quantity for a silver assay, the amount of silver, indicated as above, is to be deducted from the amount of silver obtained in the assay of any silver ore, until that quantity of litharge is consumed.

*Assay of Ores of the First Section.*—Make a preliminary assay, as already described. Suppose 10 grains of lead result; then, as 20 have furnished 10 grains, so 200 grains of ore would furnish 100 grains of lead, or 100 grains less than the quantity best adapted for cupellation; so that, referring to the assay of argol, and finding that from 30 to 32 grains reduce 200 grains of lead, then it is clear that the reducing power of from 15 to 16 grains of argol, in addition to the reducing power of 200 grains of ore, is necessary to furnish 200 grains of lead alloy. In this case, the ingredients required in the actual assay, or 'assay proper,' would stand thus:—

200 grains of ore.
200 grains of carbonate of soda.
1,000 grains of litharge.
15 to 16 grains of argol.

These materials are to be thoroughly well mixed, placed in a crucible which they about half fill, and covered first with 200 grains of common salt, and then 200 grains of borax, and submitted to the fire with the usual precautions; when the flux flows smoothly the assay is complete; it may be removed and allowed to cool, the crucible broken, and the button obtained must be hammered into a cubical form, and should approximate to 200 grains, either more or less, within 10 grains. Two crucibles must always be prepared. It will also be here convenient to mention that the argol and nitrate of potash are the only substances whose quantities vary in the assay of silver ores, the amount of these variations being determined by the preliminary or classification assay.

*Assay of Ores of the Second Section.*—If the preliminary assay of the sample submitted to assay furnish from 18 to 22 grains of lead, then the assay proper may be thus made:—

200 grains of the ore,
200 grains of carbonate of soda,
1,000 grains of litharge,

well mixed, and covered with salt and borax as above. Fuse with due care, and reserve buttons of lead alloy for cupellation.

*Assay of Ores of the Third Section.*—If the sample on preliminary assay furnished 40 grains of lead, then the 200 grains employed in assay proper would give 400 grains, or 200 grains of lead in excess: refer now to note-book for quantity of lead oxidised by nitre: suppose the nitre pure as just stated, 23 grains will oxidise 100, therefore 46 grains are equivalent to 200, and the assay proper will stand thus:—

200 grains of the ore.
200 grains of carbonate of soda.
1,000 grains of litharge.
46 grains of nitrate of potash.

The nitrate of potash is to be weighed first, finely pulverised, and then well mixed with the remaining substances, and covered with salt and borax. The crucible in this assay must be larger than in the two preceding cases; the mixture should not more than one-third fill it, as there is a considerable action set up between the oxygen of the nitre and the sulphur or arsenic, or any other substance that may be the reducing agent in the ore; for in fact the nitre does not directly oxidise the lead, which sulphur, &c., might have reduced, but oxidises its equivalent quantity of sulphur, or whatever other reducing substance there may be in the ore, so as only to leave a sufficient amount to reduce 200 grains of lead, in lieu of the 400 as indicated by preliminary assay, or when the reducing power of the ore was allowed to come into full play. The buttons obtained in this case are also to be reserved for cupellation.

*Scorification.*—Scorification has, like fusion with litharge, the effect of producing an alloy of lead capable of cupellation, and a very fusible slag composed of oxide of lead, and all the matters foreign to silver, converted into the state of oxide. In the crucible assay as just described the oxidation of these substances takes place by the action of the litharge, which furnishes at the same time by its reduction the lead necessary to form the alloy, whilst in scorification all the substances susceptible of oxidation are oxidised in the roasting by means of the oxygen of the air, and the litharge itself is produced by the oxidation of part of the lead mixed with the ore to be assayed.

In this operation vessels termed scorifiers (see p. 131) are employed. They are heated in the muffle of the cupelling furnace, and as many assays may be made at one time as the muffle holds scorifiers.

Before introducing the scorifiers into the muffle, a given weight of the ore reduced to powder is mixed intimately with a certain quantity of granulated lead, and placed in each. They must then be heated gradually for about a quarter of an hour, with the door of the muffle closed, in order to fuse the lead; then diminish the heat, and allow access of air by opening the door. The current thus established in the muffle soon causes the commencement of the roasting; and this roasting goes on without its being necessary to continually agitate the mass, as in the case of pulverulent substances.

During the oxidation, a slag is formed on the fluid metal, which is thrown towards the edges, and which, by continually augmenting, at last entirely covers the bath. This slag, which is often solid at the commencement, becomes softer and softer, and at last becomes perfectly fluid; because, in proportion to the advance of the operation, the proportion of oxide of lead continually increases. When it is judged that the scorification has been carried far enough, the melted matter is stirred with a rod of iron, in order to mix with the mass the hard or pasty parts attached to the bottom or sides of the scorifier. The fire is then urged so as to completely liquefy the slags. It may be ascertained when they are sufficiently fluid by plunging into them a red hot iron rod, which must only be covered with a slight coating, capable of running off, and not solidifying into a drop at the end.

This condition of liquidity is indispensable, in order to enable the metallic globules to unite into a single button. When this end is not attained, it is because the scorification has not been carried sufficiently far, or because a sufficient quantity of lead has not been added to form the flux, in which case a fresh quantity must be added, or, what is preferable, the assay recommenced with larger proportions.

When the operation is finished, the scorifier must be removed, and its contents immediately poured into a circular or hemispherical ingot mould (see fig. 27, p. 66). The metallic particles fall to the bottom, and as the cooling proceeds they form a button covered by the slag, which is readily detachable by a blow of a hammer: it ought to be very homogeneous and vitreous, and its colour varying from brown to greenish.

It is always advisable to examine it, and ascertain if it contain metallic globules. The button ought to be as ductile as ordinary lead; if not it cannot be cupelled, and must be submitted to a fresh operation. It is in general advantageous to push the scorification to its greatest extent, because experiment has proved that less silver is lost than when a large button is cupelled. Nevertheless, there is a limit, because if the silver-lead produced be too rich, the least loss in the shape of globules would cause a notable one in the silver. Besides, as litharge exercises a very corrosive action on earthy matters, if the scorification be continued for a great length of time it sometimes happens the vessel is pierced, and the assay has to be recommenced. The button of lead remaining ought to weigh about 200 to 300 grains, when the ores treated are of ordinary richness. The length of time a scorification takes is from half an hour to an hour. The scorifier can be rendered less permeable to the litharge by being rubbed inside with chalk, or better still red ochre.

There may be distinguished three distinct periods in the operation, viz. the roasting, the fusion, and the scorification. At first a strong fire is employed; but the doors of the furnace are opened as soon as the mixture is fused. The mineral, being specifically lighter than the lead, is then seen floating on its surface, or forming masses in it; the roasting then commences, and from the appearance of the vapours, the nature of the combustible matter it contains may be judged. Sulphur produces clear grey vapours: zinc, blackish vapours, and a brilliant white flame: arsenic, whitish-grey vapours: antimony, fine red vapours, &c. When no more fumes are seen, the mineral has disappeared, and the fused lead perfectly uncovered, the roasting has terminated: this generally requires from eighteen to twenty minutes. At this time the fire is urged, so as to cause all the substances in the scorifier to fuse. It can be asertained that the fusion is complete, by the following signs: at the instant the muffle is opened, the button becomes whitish-red with a greyish-black band, and there arise from the melted mass clear white fumes of lead, and the slag appears like a ring encircling the metal. The third period then commences: the furnace is cooled, as in the roasting, and the lead is allowed to scorify until it is entirely covered with fused oxide: this last period generally lasts about fifteen minutes. The fire is then increased for about five minutes, and the contents of the scorifier poured into the mould.

The process of scorification is applicable to all argentiferous matters, and is at the same time the most exact method of assay, as also the most convenient, when a large number of assays are required at the same time, because they are entirely executed in the muffle, which, with most assayers, is generally hot: it, however, requires a greater number of vessels—as cupels, &c.

When the silver ores are stony, the oxide of lead formed during the roasting combines with the gangue, forming a fusible compound, whilst the remaining lead alloys with the silver. When the ores are metallic, the oxidisable bodies absorb oxygen from the atmosphere; and the oxides so formed combine with the litharge produced at the same time, forming a compound which becomes very fusible in proportion as the oxide of lead increases; and if the scorification has not been pushed sufficiently far, the button will contain, besides silver and lead, a little copper, which will not, however, interfere with the cupellation. There is this one peculiarity about scorification, that however small the proportion of lead may be that is used, at the end of the operation the slag does not contain any oxysulphide. For instance, even when oxysulphides are produced in the course of scorification, they are completely decomposed in the roasting, and in consequence it is very rarely that the slag retains any proportion of silver; and as to the proportion of lead employed, only just enough to render the slag liquid, and to produce sufficient lead for cupellation, is necessary.

It is different, however, when the sulphides and arseniosulphides are assayed by means of litharge; for from 30 to 50 parts of that substance must be employed to prevent the scoriæ retaining any silver, or, as already pointed out, a certain proportion of nitre must be added.

All scorifications may be conducted by the simple addition of lead; but it has been proved that the operation proceeds more quickly, and with less danger to the scorifier, when borax is employed. This salt dissolves the oxides in proportion as they are produced, as also the gangues, and forms a very liquid slag from the commencement of the operation, which does not happen when lead alone is used, because litharge, which can alone cause the fusion, is only present in the slag in sufficient proportion at a very advanced stage of the operation.

When the slag is liquid at the beginning of the operation (as occurs in the use of borax), it is continually thrown on the sides of the scorifier, and forms a ring on the surface of the bath, leaving in the centre the metallic substance, having a considerable extent of surface, which is continually diminishing.

The current of air, being thus directly in contact with the fused metals, rapidly causes their oxidation, which does not take place when the semifluid substances float here and there on the metallic bath. The proportion of lead and borax necessary for a scorification varies exceedingly, according to the nature of the substance under assay, and ought to be greater in proportion as the substances, or resulting oxides, are difficult of fusion. In ordinary cases 12 parts of lead, and 1 of glass of borax, are employed; but sometimes 32 of lead, and 3 of borax, are required. A large proportion of borax is useful, especially when the substances contain much lime, oxide of zinc, or oxide of tin.

Instead of borax, glass of lead may be employed. It acts as a flux on silica; but its action is much less effective than that of borax.

There are some substances which scorify with a small proportion of lead. Thus, for galena and sulphide of copper, 2 parts of lead suffice; but 8 parts are required for ores which contain much gangue.

Antimonide of silver can be scorified with 8 parts of lead, but according to experiments made in the Hartz, it appears that the slag retains about $\frac{1}{140}$th of silver; with 16 parts of lead $\frac{1}{200}$th of fine metal is still lost; but with 3 of borax and 16 of lead not the slightest trace remains in the slag.

It is very difficult to separate tin and silver by the dry way. The best method is to roast the alloy in a scorifier, adding to it 16 parts of lead and 3 of borax at least, and operating as before described.

Speiss almost always contains silver, and is one of the most difficult substances to assay. If nickel be present, the button cannot be cupelled. Generally, speiss may be scorified with 16 parts of lead; and the same operation is gone through twice or thrice, adding each time a fresh quantity of lead. The operation would probably succeed by roasting the speiss in the scorifier before adding the lead.

*Special Instructions for the Scorification Assay of Ores of the First Class.*—This mode of assay has an advantage over the crucible assay just described, inasmuch as if properly conducted no preliminary assay is required: but this is greatly counterbalanced by the fact that not more than 50 grains of ore can be operated on in one scorifier, and that good or trustworthy results cannot be obtained by this method unless four scorifiers are employed for each assay, so that in all 200 grains of ore may be employed. There are thus employed four scorifiers to three crucibles, and four cupels to two cupels; as in one case four buttons are to be submitted to cupellation, and in the other only two. When very rich copper ores, however, have to be assayed for silver, the plan by scorification is very useful, as in the crucible operation much copper is reduced with the lead, so as to require a very large quantity of lead for its conveyance as oxide into the cupel. This class of assay will, however, be particularly noticed under the head Assay of the Alloys of Silver.

*Assay in Scorifier.*—Weigh out 300 grains of granulated lead, place them in a scorifier, then add 50 grains of pulverised fused borax, and 50 grains of the ore to be assayed, well mix them in the scorifier by aid of a spatula, and cover the mixture with other 300 grains of granulated lead: prepare in this way four scorifiers, place them in the muffle with the tongs (*b*, fig. 26, page 65,) and carefully watch them with all the precautions before pointed out: when the surface of the metal is quite covered with fused oxide, pour the contents of each scorifier into one of the hollows of the mould depicted at fig. 27, page 66. When the mass of slag and metal is cold, separate the latter from the former by means of the hammer and anvil, hammer the metal into the form of a cube, and reserve it for cupellation.

*Assay of Substances of the First Class admixed with Native or Metallic Silver.*—The same kind of calculation is necessary in the assay of ores as above, as in the case of copper ores containing metallic copper. The sample must be carefully weighed. Suppose it to weigh 2,500 grains. It must be pulverised, and as much as possible passed through the sieve with eighty meshes to the linear inch. It will be thus divided into two parts: the one passing through the sieve is mineralised silver—that is, silver ore of various kinds mixed with earthy matter, and a very small quantity of metallic silver which has been sufficiently divided to pass through a sieve of such a degree of fineness; the other, impure metallic silver, which has been unable to pass through the sieve. The weights of both portions are carefully taken, and thus noted—

| | | |
|---|---|---|
| **Rough metallic silver** . . . . | 5·07 grs. | 78.27[illegible] |
| **Ore through sieve** . . . . . | 2494·93 „ | |
| **Total weight of sample** . . . . . | 2500·00 „ | |

Assay the ore which passed through the sieve as already directed, and the rough silver as directed under the head Assay of Silver Alloys. Note the quantity of silver obtained in each experiment. Thus: suppose 200 grains of ore yielded 2 grains of fine silver, and the 5·07 grains of rough silver 4 grains of fine silver by cupellation, the number of ounces of fine silver in the ton is thus calculated.

On referring to Table III. in Appendix, it will be found, that if 200 grains of ore yield 2 grains of fine silver, 1 ton will yield 326 oz. 13 dwts. 8 grs. of fine silver; so that the average produce of the ore is the above amount.

Then, if 5·07 grains of rough silver yield 4 grains of fine silver, 200 grains would yield, by calculation, 159·763 grains of fine silver.

Thus—

$$\frac{200\times 4}{5\cdot 07}=159\cdot 763$$

Now, by referring to Table III. in the Appendix, it will be found that 200 grains of ore give .159 grains of fine silver=25, 970 ounces per ton: and that 200 grains of ore give ·763 grains of fine silver=124 ozs. 12 dwts. 11 grains: therefore, the 5·07 grains of rough silver contain after the rate of 26,094 ozs. 12 dwts. 11 grs. per ton, thus—

25,970 ozs.+124 ozs. 12 dwts. 11 grs.=26,094 ozs. 12 dwts. 11 grs.

Thus we have—

| | ozs. | dwts. | grs. |
|---|---|---|---|
| Average produce of ore . . . . | 326 | 13 | 8 |
| Average produce of rough silver . . | 26,094 | 12 | 11 |

per ton of 20 cwts.

Then, as in the case of the copper, multiply the weight and produce of each portion together, add the resulting total products, and divide the sum by the weight of the sample. For this purpose it is better to reduce the pennyweights and grains to their decimal values. Thus 13 dwts. 8 grs. is nearly equal to ·67 of an ounce, and 12 dwts. 11 grs. to ·62 of an ounce; therefore the quantities above will stand thus —326·67 ozs., and 260,94·62 ozs.

Then 326·67 × 2494·93=815018·7831
and 26094·62 × 5·07=132296·7234
and $$\frac{815018\cdot 7831+132299\cdot 7234}{2500}=378\cdot 9\text{ oz.}$$

or 378 ozs. 18 dwts. (nearly) per ton of the original sample, before pulverising and sifting.

In every case of assay yet described, it may be mentioned that if the sample contained gold, the whole of that metal will be found with the silver, as obtained by cupellation, and may be separated as stated under the head Gold Assay.

*Cupellation.*—Cupellation is one of the most ingenious operations that can be imagined; it has been known from time immemorial, has many characters in common with scorification, and is effected in nearly the same manner. Like that, it has for its end the separation of silver and gold from different foreign substances, by means of lead; but it differs in this, that the scoriæ produced are absorbed by the substance of the vessel named a cupel, in which the operation is made, instead of remaining on the melted metal, the latter remaining uncovered and in contact with the air, so that the extraneous metals are not only oxidised, but also all the lead; and there remains nothing but the pure metals, silver and gold, or an alloy of them in the cupel.

Cupellation requires, as an indispensable condition, that the slag should have the property of penetrating and soaking into the body of the substance forming the cupel; it is, therefore, only applicable to a certain number of substances, and not to all, like scorification. The oxides of lead and bismuth, in a state of purity, are the only oxides which possess the property of soaking into the cupel; but by the aid of one or the other, various oxides which by themselves form infusible scoriæ on the cupel, acquire the property of passing through it: therefore, on making a cupellation, it is necessary to fuse the substance with a sufficient proportion of lead or bismuth, so that the oxides they produce may combine with the oxides of all the foreign metals produced in the operation, and carry them into the body of the cupel.

This proportion varies with the nature of the substances cupelled, and other circumstances. The quantity required in ordinary cases will be mentioned hereafter.

The cupels or porous vessels in which the operation is made, ought to have a sufficiently loose texture to allow the fused oxides to penetrate them easily, and at the same time to possess sufficient solidity to enable them to bear handling without fracture; and, moreover, they ought to be of such a nature as not to enter into fusion with either oxide of lead or bismuth. For a description of their mode of manufacture, see page 128.

The following is the method in which an ordinary cupellation is conducted :—The furnace being heated, the bottom of the muffle is covered with cupels, placing the largest towards the end; and if they are required to be heated as quickly as possible, they may be placed upside down, and turned, at the instant of use, by means of the tongs. When the interior of the muffle is reddish-white, the matters to be cupelled may be introduced. When the cupels have been placed in their proper position, great care must be taken from the commencement to blow out of them all cinders, ashes, and other extraneous substances which may have fallen into them.

The substance to be cupelled is sometimes an alloy, which can pass without addition, and sometimes a compound, to which lead must be added. In the first case, the alloy is laid hold of by a small pair of forceps, and deposited gently in the cupel. In the second case, the substance to be cupelled is enveloped in a sheet of lead of suitable weight, and placed, as before, in the cupel; or the necessary quantity of lead may be first placed in the cupel, and when the lead is fused, the substance to be cupelled added, taking care not to agitate the melted mass, and cause loss by splashing. If the substance to be cupelled is in very small pieces, as grains or powder, it must be enveloped in a small piece of blotting-paper, or, still better, in a piece of very thin sheet lead, giving it a slightly spherical form, and dropping it gently into the mass of molten metal in the cupel. Sometimes the substance is gradually added, by means of a small iron spoon; but it is preferable to use paper, or thin lead, as just recommended.

When the cupels are filled, the furnace is closed, either by the door or by pieces of lighted fuel, so that the fused metals may become of the same temperature as the muffle. When this point has been gained, air is allowed to pass into the furnace; the metallic bath is then in the state termed *uncovered*; that is, it presents a convex surface, very smooth and without slag. When the air comes in contact with it, it becomes very lustrous, and is covered with luminous and iridescent patches, which move on the surface, and are thrown towards the sides. These spots are occasioned by the fused oxide of lead which is continually forming, and which, covering the bath with a very thin coating of variable thickness, presents the phenomenon of coloured rings.

The fused litharge, possessing the power of moistening (so to speak) the cupel, is rapidly absorbed by it when sufficiently porous, so that the metallic alloy is covered and uncovered every instant, which establishes on its surface a continual motion from the centre to the circumference. At the same time a vapour rises from the cupels which fills the muffle, and is produced by the vapour of lead burning in the atmosphere. An annular spot is soon observed on the cupel around the metal, and this spot increases incessantly until it has reached the edges.

In proportion as the operation proceeds, the metallic bath of silver-lead diminishes, becoming more and more rounded; the shining points with which it is covered become larger and move more rapidly; lastly, as the whole of the lead separates, the button seems agitated by a rapid movement, by which it is made to turn on its axis; it becomes very lustrous, and presents over its whole surface all the tints of the rainbow: suddenly the agitation ceases, the button becomes dull and immovable, and after a few instants it takes the look of pure silver. This last part in the operation of cupellation is termed the *brightening*, *fulguration*, or *coruscation*.

If the button be taken from the muffle directly after the brightening, it may throw off portions of its substance; this must be avoided, especially when the button is large. The button, when covered by mammillated and crystalline asperities, is said to have 'vegetated.' The cause of this effect seems to be, that when the fused buttons are suddenly exposed to the cold air, the silver solidifies on the surface, whilst that in the interior remains liquid. The solid crust, contracted by cooling, strongly compresses the liquid interior, which opens passages for itself, through which it passes out, and around which it solidifies when in contact with the cool air. But it sometimes happens that, when the contraction is very strong, a small portion of the silver is thrown off in the shape of grains, which are lost.

After brightening, the cupels must be left for a few minutes in the furnace, and drawn gradually to the mouth, before they are taken out, so that the cooling may be slow and gradual. These precautions are nearly superfluous when the buttons are not larger than the head of an ordinary pin.

As silver is sensibly volatile, it is essential, in order that the smallest possible quantity be lost, to make the cupellation at as low a temperature as may be. On the other hand, the heat ought to be sufficiently great, so that the litharge may be well fused and absorbed by the cupel; and, moreover, if the temperature be too low, the operation lasts a very long time, and the loss by volatilisation will be more considerable than if the assay had been made rapidly at a much higher temperature.

Experience has proved that the heat is too great when the cupels are whitish, and the metallic matter they contain can scarcely be seen, and when the fume is scarcely visible and rises rapidly to the arch of the muffle. On the contrary, the heat is not strong enough when the smoke is thick and heavy, falling in the muffle, and when the litharge can be seen not liquid enough to be absorbed, forming lumps and scales about the assay. When the degree of heat is suitable the cupel is red, and the fused metal very luminous and clear.

In general, it is good to give a strong heat at the commencement, so as to well uncover the bath, then to cool down, and increase the heat at the end of the operation for a few minutes, in order to aid the brightening. There can be no inconvenience in urging the temperature at first, because the silver-lead is then poor, and much precious metal cannot be lost by volatilisation. The increase of fire given towards the end is for the purpose of separating the last traces of lead, from which it is very difficult to free the silver; but this strong fire must not be continued long, otherwise there might be a notable loss by volatilisation. When the assay of very poor argentiferous matters is made, the heat can be kept up nearly all through the cupellation. It generally succeeds better when the temperature is too high than too low.

The force of the current of air which passes through the muffle is another very important thing in the success of the operation. Too strong a current cools the cupel, oxidises too rapidly, and the assay would be spoilt. With a too feeble current the operation proceeds slowly, the assay remains a long time in the fire, and much silver is lost by volatilisation.

When the litharge is produced more rapidly than it can be absorbed by the cupel, or when it is not liquid enough, which may happen from the furnace being too cold, or when other oxides, produced at the same time, diminish its fusibility, it accumulates gradually on the fluid metal, forming, at first, a ring which envelopes its circumference, and which gradually extending, covers the whole surface: at this period the assay becomes dull, and all movement ceases. When the operation is carefully attended to, it is nearly always possible to avoid this accident. If, at the first moment, any signs are manifested of this evil, the temperature of the muffle must be raised, either by shutting the door, or placing in it burning fuel: the assay will, in a little time, resume its ordinary course. But when the cause of the mishap is supposed to be the abundance of foreign oxides in the assay, a fresh proportion of lead must be added.

It can be ascertained whether an assay has *passed* well by the aspect of the button. It ought to be well rounded, white, and clear, to be crystalline below, and readily detached from the cupel. When it retains lead, it is brilliant below and livid above, and does not adhere at all to the cupel.

In order to detach the button, seize it with a strong pair of pliers (see fig. 80), and examine with a microscope (see fig. 81), brushing it to detach small particles of litharge which may adhere to it, and place it in the pan of a balance (fig. 13, page 24) which will indicate the $\frac{1}{1000}$th of a grain. The weight of the silver furnished by the lead or litharge employed in the operation ought to be subtracted from the amount of silver obtained; so that it is necessary to ascertain the richness of these matters beforehand, as they are never completely free from silver. The poorest of them contain from $\frac{1}{100000}$th to $\frac{1}{10000}$th.

Fig. 80.

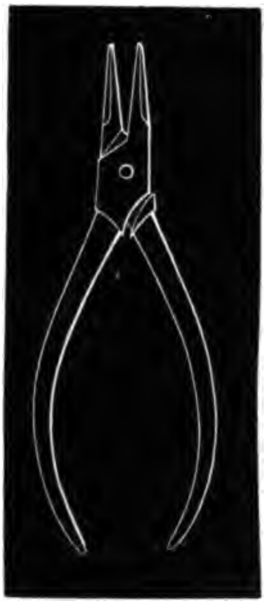
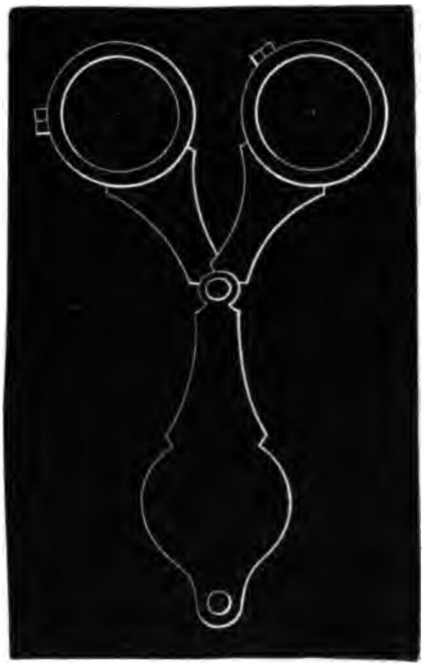


Fig. 81.



Sometimes an equal quantity of lead is placed in another cupel, and the silver thus obtained placed in the balance pan containing the weights.

Cupellation does not give the exact proportion of silver contained in an alloy. There is always a loss, and this loss is always greater than that which takes place in the large way, as in the latter process a greater quantity is always obtained than that determined by the assay. The loss of silver is traceable to three causes; 1st, volatilisation; 2ndly, to oxidation; 3rdly, and lastly, to the absorbtion of minute globules of silver into the body of the cupel. It is certain volatilisation takes places, because a notable quantity of silver is always found deposited on the sides of the furnace and chimney in the shape of dust; and silver, which is volatile by itself, becomes much more so when alloyed with lead, and is carried away by the vapours of the latter, and found in the pulverulent deposits, termed *lead smoke or fume*, which proceeds from the combustion of the latter metal in the air. Nevertheless, this cause of loss is not very important, for it is rare that the fume contains more than $\frac{1}{10000}$th of silver, and accurate experiments have proved that in cupellation in the small way not more than two to three per cent. of lead is volatilised. It is certain that a portion of the silver found in cupels which have been used for assays exists in the state of oxide, for no part of their mass is free—it is found even in the bottom: besides it is known that the carbonate of lead precipitated from acetate of lead made from litharge contains silver, and a notable quantity of that metal is found even in the sulphate of lead prepared by means of alum from the acetate (excepting the sulphate is repeatedly washed with water).

It has been remarked that the centres of cupels which have been used for assays are richer in silver than the parts nearer the circumference, and that under the button there is a spot of bright yellow, which appears to be oxide of silver. But the most important cause of loss in an assay is the property which the alloys of silver and lead possess of introducing themselves into the pores of the cupel. The quantity thus lost is in proportion to the coarseness of the cupel. For the same quantity of silver, the loss which takes place in an assay varies according to the nature of the alloy, and the circumstances under which the assay is made; so that it is not possible to form accurate tables of correction. This loss is much augmented with the quantity of lead employed, but without its being proportionate; so that when scorification is had recourse to it is advantageous to continue the operation for some length of time, in order that the metallic button may be reduced to the smallest suitable volume.

In the assay of rich alloys, the proportion to the total amount of silver is very small, but notable; and it has been calculated for the alloys of copper employed in the arts at $\frac{1}{3000}$th; but in the assay of poor ores, such as galena and other minerals treated in the large way, the loss is very great, for it is usually as high as $\frac{1}{500}$th.

By extracting the lead from cupels used in this class of assay, the metal furnished contains from about $\frac{1}{300000}$th to $\frac{1}{500000}$th of silver. The following experiment will give an idea of the influence of the proportion of lead on the loss of silver: 100 grains of commercial litharge were fused with 10 grains of black flux, and gave 27 grains of lead, and a slag; this was pulverised and reduced in the same crucible with 15 grains of black flux, and a second button was produced weighing 45 grains. These two buttons being cupelled separately, gave, the first ·0035 and the second ·001 only of silver. Three new quantities of 100 grains of the same litharge were fused; the first with ½ a part of starch, the second with 2½, and the third with 10 of the same reducing agent. The resulting buttons of lead weighed respectively 5·28 and 7·9 grains. These buttons were cupelled, and furnished ·0035, ·0035, and ·003 respectively. From these experiments it will be seen that when the litharge is not reduced completely, there remains a notable proportion of silver in the scoriæ; but, nevertheless, in order to extract the largest possible quantity, the whole must not be reduced. Indeed, but a twentieth part need only be reduced, because more precious metal is lost in the cupellation of a large quantity of lead than remains in the portion not reduced. The loss of silver in large cupellations is less than that which takes place in an assay, because in the large way the litharge, or the greater part of it, is run off; whilst in an assay the cupel totally absorbs it, so that the latter presents, relatively to the same mass of lead, a very much smaller surface in the large than in the small way: now it can be readily seen that the quantity of silver lost by absorption into the pores of the cupel must be proportioned to its surface, all things being equal.

It has been ascertained by experiment that a cupel absorbs about its own weight of litharge; so that from this fact a cupel of the proper size may be chosen, when the weight of lead to be cupelled is ascertained. It is always better to have the cupel about ⅓ or ¼ as heavy again as the lead to be cupelled.

The various metals found in an alloy, which can be submitted to cupellation, scorify in proportion to their oxidisability. Those most oxidisable scorify with the greatest rapidity, and vice versa; so that those which have the greatest affinity for oxygen accumulate in the first portions of litharge formed, which, by that means becoming less fusible, sometimes lose the property of penetrating the cupel; hence the reason why cupellations always present more difficulties at the commencement of the operation than towards the end when the litharge formed is nearly pure oxide of lead, and can contain only oxide of copper.

The appearance of the cupel used in an assay will give indications of the metals the alloy contained. Pure lead colours the cupel straw-yellow, verging on lemon-yellow. Bismuth, straw-yellow passing into orange-yellow. Copper gives a grey, dirty red, or brown, according to its proportion. Iron gives black scoriæ, which form at the commencement of the operation, and are generally found at the circumference of the cupel. Tin gives a grey slag. Zinc leaves a yellowish ring on the cupel, producing a very luminous flame, and occasioning losses by carrying silver in its vapour, and by projecting it from the cupel in its ebullition. Antimony and sulphate of lead in excess give litharge-yellow scoriæ, which crack the cupel; but, when not produced in too great a proportion, are gradually absorbed by the litharge. If the lead alloy submitted to cupellation is found to produce this effect, a fresh portion must be mixed with its own weight of lead and scorified: the button so obtained can now be cupelled.

*Amalgamation.*—There are a certain number of argentiferous matters which can be assayed by amalgamation, as they are treated in the large way by that method. Amongst these are native silver chlorides, sulphides, and arseniosulphides, which contain neither lead nor copper.

But this process is seldom had recourse to, because it is long, troublesome, and less exact than those just described.

### *Substances of the Second Class.*

Native silver.
Alloys of copper and silver.
Alloys of other metals and silver (artificial).
Antimonide of silver.
Arsenide of silver.
Telluride of silver (AgTe).
Auriferous telluride of silver (see gold).
Hydrargyride of silver (amalgam), ($Hg^2Ag$).
Auride of silver (see gold).

The following method of separating silver from galena is given in the ‘Chemical News,’ vol. ii. p. 239.

‘Galena consists, as is well known, of the sulphide of lead, mixed with a variable proportion of the sulphide of silver, and both these substances fuse together, or melt at a bright red heat. Now, it so happens that, when sulphide of silver is fused with chloride of lead, what is called a double decomposition takes place; that is to say, chloride of silver and sulphide of lead are formed. Consequently, if we fuse together a quantity of argentiferous galena and chloride of lead, we shall remove the whole of the silver from the galena, and replace it by sulphide of lead. This, then, is the process: mix together the galena and chloride of lead in the proportion of 100 lbs. of galena, 1 lb. of chloride of lead, and 10 lbs. of chloride of sodium or common salt; or, if the galena be very argentiferous, add a larger amount of chloride of lead. The whole is then fused together, when the chloride of silver and common salt rise to the surface, and may be skimmed off, and the desilverised galena falls and may be run out from the bottom. The mixture of chloride of silver and salt may then be decomposed by lime and charcoal, or in any other manner, so as to reduce the silver and a portion of the surplus chloride of lead, by which a metallic mass will result, suitable for the operation of the cupel.’

*General Remarks on the Assay of the Alloys of Silver and Copper.*—The assay of these alloys is nearly always accomplished (at least in England) by cupellation. This assay is most important, as it is by the results obtained in the manner hereafter described that the price or value of all kinds of silver bullion is determined.

This class of cupellation is effected without difficulty, because the oxide of copper forms so slowly, that the litharge is always enabled to pass it into the body of the cupel. After having weighed the lead and placed it in the cupel, as soon as it is perfectly fused place in it the alloy to be assayed, wrapped either in blotting-paper or thin leaf-lead. It is essential, in this class of assay, to employ a sufficient quantity of lead to carry away all the copper. We may always be sure of succeeding, whatever the alloy may be, by employing the maximum proportion of lead, that is to say, the quantity necessary to pass pure copper; but as the loss which the silver undergoes increases with the length of the operation and with the mass of the oxidised matters, it is indispensable to reduce this loss as much as possible by reducing the proportion of lead to that which is strictly necessary. Long experience has proved that silver opposes the oxidation of copper by its affinity, so that it is necessary to add a larger amount of lead in proportion to the quantity of silver present.

M. D'Arcet has obtained the following results by the most accurate experiments:—

| Standard of silver | Quantity of copper alloyed | Quantity of lead necessary | Relation of lead to copper |
|---|---|---|---|
| 1000 | 0 | $\frac{3}{10}$ths | |
| 950 | 50 | 3 | 60 to 1 |
| 900 | 100 | 7 | 70 — 1 |
| 800 | 200 | 10 | 50 — 1 |
| 700 | 300 | 12 | 40 — 1 |
| 600 | 400 | 14 | 35 — 1 |
| 500 | 500 | 16 to 17 | 32 — 1 |
| 400 | 600 | 16 — 17 | 27 — 1 |
| 300 | 700 | 16 — 17 | 23 — 1 |
| 200 | 800 | 16 — 17 | 20 — 1 |
| 100 | 900 | 16 — 17 | 18 — 1 |
| pure copper | 1000 | 16 — 17 | 16 — 1 |

It is remarkable that below the standard of 500, the same proportion of lead must be employed, whatever that of copper. This fact is repeatedly verified by experiment. Whenever fine silver is fused in a cupel, it is always necessary to add lead, in order to cause the button to unite and form well. If less than $\frac{3}{10}$ths of lead be employed, the button will be badly formed; the litharge cannot separate but by the action of a very strong heat, and a considerable loss of silver ensues. If, on the contrary, $\frac{3}{10}$ths of lead is exceeded, the cupellation goes on well, but the loss is greater on account of the duration of the process. These proportions also ought to vary with the temperature. M. Chaudet has found, that to cupel an alloy containing $\frac{900}{1000}$ths of silver, 5 parts of lead are required in the middle of the muffle, 10 in the front, and only 3 at the back.

The proportion of copper carried off by litharge varies not only with the temperature, but even for the same temperature in relation to the amount of copper and lead the alloy contains. By cupelling 100 parts of copper with different proportions of lead in the same furnace, M. Karsten obtained the following results:—

| Lead added | Copper remaining after cupellation | Quantity of lead consumed in carrying off 1 of copper |
|---|---|---|
| 100 | 78·75 | 3· |
| 200 | 70·12 | 7·1 |
| 300 | 60·12 | 7·7 |
| 400 | 49·40 | 7·9 |
| 500 | 38·75 | 8·1 |
| 600 | 26·25 | 8·15 |
| 700 | 19·75 | 8·00 |
| 800 | 8·75 | 8·70 |
| 900 | 5·62 | 9·50 |
| 1000 | 1·25 | 10·10 |
| 1050 | 0·00 | 10·50 |

From which we see that the lead carried away from $\frac{1}{15}$th to $\frac{1}{10}$th of its weight of copper. Much less lead can be employed in a cupellation by making the alloy maintain its richness of copper throughout the operation. This can be accomplished by adding to the alloy in the cupel small doses of lead, in proportion as that first added disappears by oxidation. If, for example, an alloy composed of 4 parts of copper and one of silver be fused with 10 of lead, by adding successive small doses of the latter, as already pointed out, but 7 parts will be consumed, although in the regular way from 16 to 17 would be employed.

The proportion of oxide of copper contained in the litharge increases each instant, and goes on incessantly increasing when an alloy of copper and lead is cupelled which contains an excess of copper. According to M. Karsten, this proportion is always about 13 per cent. at the commencement, and 36, or more than a third, at the end of the operation.

In the assay of the coined alloys of copper and silver, the loss of silver may even amount to five thousandths; but the loss is variable, and is proportionally greater as the standard of the alloy is lower.

The following Table contains the results of many experiments made on this subject:—

| Exact standard | Standard found by cupellation | Loss, or the quantity of fine metal to be added to the standard as obtained by cupellation |
|---|---|---|
| 1000 | 998·97 | 1·03 |
| 975 | 973·24 | 1·76 |
| 950 | 947·50 | 2·50 |
| 925 | 921·75 | 3·25 |
| 900 | 896·00 | 4·00 |
| 875 | 870·93 | 4·07 |
| 850 | 845·85 | 4·13 |
| 825 | 820·78 | 4·22 |
| 800 | 795·70 | 4·30 |
| 775 | 770·59 | 4·41 |
| 750 | 745·38 | 4·52 |
| 725 | 720·36 | 4·64 |
| 700 | 695·25 | 4·75 |
| 675 | 670·27 | 4·73 |
| 650 | 645·29 | 4·71 |
| 625 | 620·30 | 4·70 |
| 600 | 595·32 | 4·68 |
| 575 | 570·32 | 4·68 |
| 550 | 545·32 | 4·68 |
| 525 | 520·32 | 4·68 |
| 500 | 495·32 | 4·68 |
| 475 | 470·50 | 4·50 |
| 450 | 445·69 | 4·31 |
| 425 | 420·87 | 4·13 |
| 400 | 396·05 | 3·95 |
| 375 | 371·39 | 3·61 |
| 350 | 346·73 | 3·27 |
| 325 | 322·06 | 2·94 |
| 300 | 297·40 | 2·60 |
| 275 | 272·42 | 2·58 |
| 250 | 247·44 | 2·56 |
| 225 | 222·45 | 2·55 |
| 200 | 197·47 | 2·55 |
| 175 | 173·88 | 2·12 |
| 150 | 148·30 | 1·70 |
| 125 | 123·71 | 1·29 |
| 100 | 99·12 | 0·88 |
| 75 | 74·34 | 0·66 |
| 50 | 49·56 | 0·44 |
| 25 | 24·78 | 0·22 |

These numbers, however, are not constant, and vary with the circumstances under which the assays are made: two assays made from the same ingot, by the same assayer, can differ as much as four or five thousandths. Tillet has remarked that the cupels can retain double as much silver as is lost; which proves, as has already been mentioned, that the silver obtained by cupellation is not perfectly pure, but may retain as much as 1 per cent. of lead.

### *Special Instructions for the Assay of the Alloys of Silver and Copper.*

As before stated, peculiar weights are employed in the assay of silver bullion; and the silver assay pound, with its divisions, will be found described at page 31.

In the 'General Remarks on the Assay of the Alloys of Silver and Copper,' it will be seen that the alloy must be cupelled with a quantity of lead, varying with the amount of copper present in the alloy. Standard silver cupels very well with five times its weight of lead; but when the approximative quantity of alloy present is not known, it must be determined by a preliminary assay.

*Assay for Approximative Quantity of Alloy.*—Weigh off 50 grains of pure or test lead; place them in a cupel previously made red-hot; when the lead is fused, and its surface covered with oxide, place in it by means of the light tongs (*a*, fig. 26, page 65) 2 grains of the alloy under assay, wrapped in a small piece of thin paper. Allow the cupellation to go on according to the instructions, and with all the precautions already given, and when complete, weigh the resulting button, and, according to its weight, add lead in the actual assay in the quantity that is sufficient, as exhibited in the Table at page 488.

*Assay Proper of Silver Bullion.*—In this assay the operator requires silver known to be standard, and pure lead. With the possession of the above substances the assay is thus proceeded with:—Place the 12 grains weight=1 lb., in the scale pan, and exactly counterbalance it with standard silver. This is to serve as a check. Remove the weight, and in its place add so much of the alloy to be assayed that the balance is again equal. In one cupel, that destined to receive the check sample, place 60 grains of lead; and in another cupel place such a number of grains of lead as may be found necessary by the preliminary assay. When the lead in both cupels is fused, add the silver alloy, and cupel with the necessary precautions. When the buttons in the cupels are cold, seize them with the pliers, and if necessary cleanse them with a hard brush, and place one in each balance pan. If they exactly balance each other, the alloy operated on is standard silver; if, however, it weighs less than the button produced from the check sample by the weight equivalent to 2 pennyweights, then it is 2 pennyweights worse than standard: on the other hand, if it be heavier by the same weight, it is 2 pennyweights better than standard. Silver is also reported as so much fine: thus, standard silver may be reported as 11 ounces 2 pennyweights fine, and so on. In case extreme accuracy be required, correction must be made according to the standard as shown by the Table at page 490. The standard silver in England is $\frac{975}{1000}$ fine.

*Assay of Alloys of Copper and Silver.*—In the treatment on the large scale of copper ores containing silver, the contained silver is found alloyed with the copper, and it often falls under the assayer's province to determine the quantity of precious metal. An assay of this kind is most conveniently accomplished by scorification before cupellation, thus: —Prepare four scorifiers; weigh into each of them 50 grains of the alloy, 50 grains of fused borax, and 600 grains of lead, and proceed as already described under the head 'Assay of Ores of the First Class by Scorification.' When the four buttons of lead are obtained, place them together in another scorifier, and submit to the furnace until the contents of the scorifier are completely covered with oxide; pour as usual, and cupel the resulting mass of lead.

*Alloys of Platinum and Silver.*—If any substance containing platinum as well as silver were assayed as already described, the button resulting from the cupellation would, in addition to the silver, contain the whole of the platinum.

In such a case the button so obtained must be thus treated:—

If the alloy contain much platinum, it must be fused with twice its weight of silver; then treated with hot nitric acid; evaporate the solution nearly to dryness; add water and hydrochloric acid, until no further precipitation of silver as a white curdy precipitate (chloride of silver) takes place. The chloride of silver may be collected either on a filter or by decantation. The solution containing the platinum is treated with excess of sal-ammoniac solution until no further precipitation takes place; the solution evaporated to dryness. When cold, dilute alcohol is added; and the insoluble yellow matter (ammonio-chloride of platinum) collected on a filter, washed with alcohol, dried, and ignited. The ignited residue is metallic platinum, which is weighed. The loss of weight which the alloy from cupel has sustained represents the amount of silver previously alloyed with it.

*Alloy of Platinum, Silver, and Copper.*—Treat such an alloy as above; and the liquid, filtered from the ammonio-chloride of platinum, will contain the copper. Acidulate it with hydrochloric acid, add metallic zinc, and proceed as directed under the head 'Humid Copper Assay.'

*Native Silver, Rough Silver left on Sieve during Pulverisation of Silver Ores of First Class, and Native Alloys of Silver—as Antimonides, &c.*—are treated by scorification and cupellation in precisely the same manner as just described for alloys of copper and silver.

Dr. W. Dyce proposed, in 'Tilloch's Philosophical Magazine' for 1805, the following process for separating gold and silver from the baser metals:—

'Hitherto the process has always been, as far as I have understood it, attended with considerable difficulty in the execution; but, by that which I am about to describe, is done with exact certainty. It was discovered and communicated to me by a gentleman in the neighbourhood. The process consists in mixing not less than two parts of powdered manganese with the impure or compound metal, which should be previously flattened or spread out so as to expose as large a surface as possible, and broken or cut into small pieces for the convenience of putting the whole into a crucible, which is then to be kept in a sufficient heat for a short time. On removing the whole from the fire, and allowing it to cool, the mixture is found to be converted into a brownish powder, which powder or oxide is then to be mixed with an equal proportion of powdered glass, and then submitted in a crucible to a sufficient heat, so as to fuse the whole; when the perfect metals are found at the bottom in a state of extreme purity, a circumstance of no small importance to the artist and the chemist, the latter of whom will find no difficulty in separating the one from the other with so little trouble compared with the usual processes, that I have no doubt it will always be practised in preference to the cupel.'

*Assay of Silver Bullion by the Wet Method.*—From that which has been stated under the head of 'Cupellation,' it will be observed that there are many sources of error; such as volatliisation of the precious metal, its oxidation in the presence of excess of oxide of lead and atmospheric oxygen, and lastly, its absorption into the body of the cupel either as oxide or metal, or in both states. These losses, as before stated, vary with the temperature, the amount of lead employed, and the texture of the cupel; and, as may be seen from the table of corrections as drawn up by D'Arcet, give a very erroneous assay, unless the addition necessary for each standard be made.

Considerable attention was called to this matter in France some years since, and a Special Commission was appointed to examine the subject thoroughly, and, if possible, to devise some means of assay which might be both easy and accurate. The result of this examination was the invention of a process of assay at once elegant and trustworthy: and as a full account of this method has not, to the author's knowledge, been translated and published in this country,* he has prepared the present from M. Gay-Lussac's Report, which formed a part of a communication from M. Thiers to Earl Granville, and which appeared in the original language in the year 1837, in a Report on the Royal Mint.

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* Some portion of this report has been published in Dr. Ure's Dictionary of Arts, Mines, and Manufactures.

The new process of assay about to be described consists in determining the fineness of silver bullion by the quantity of a standard solution of common salt ($NaCl$) necessary to fully and exactly precipitate the silver contained in a known weight of alloy. This process is based on the following principles:—

The alloy, previously dissolved in nitric acid ($NO_5$), is mixed with a standard solution of common salt, which precipitates the silver as chloride, a compound perfectly insoluble in water, and even in acids.

The quantity of chloride of silver precipitated is determined not by its weight, which would be less exact and occupy too much time, but by the weight or volume of the standard solution of common salt necessary to exactly precipitate the silver previously dissolved in nitric acid.

The term of complete precipitation of the silver can be readily recognised by the cessation of all cloudiness when the salt solution is gradually poured into that of the nitrate of silver. One milligramme of that metal is readily detected in 150 grammes of liquid; and even a half or a quarter of a milligramme may be detected, if the liquid be perfectly bright before the addition of the salt solution.

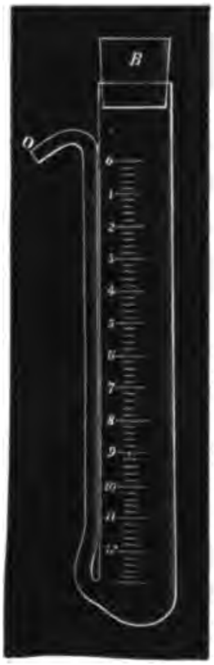
By violent agitation during a minute or two, the liquid, rendered milky by the precipitation of chloride of silver, becomes sufficiently bright after a few moments' repose to allow of the effect of the addition of half of a milligramme of silver to be perceptible. Filtration of the liquid is more efficacious than agitation; but the latter, which is much more rapid, generally suffices. The presence of copper, lead, or any other metal, with the exception of mercury (the presence of the latter metal requires a slight modification of the process, which will be hereafter pointed out), in the silver solution, has no sensible influence on the quantity of salt required for precipitation: in other words, the same quantity of silver, pure or alloyed, requires for its precipitation a constant quantity of the standard salt solution.

Supposing that 1 gramme of pure silver be the quantity operated on, the solution of salt required to exactly precipitate the whole of the silver ought to be of such strength that, if it be measured by weight, it shall weigh exactly 100 grammes, or if by volume 100 cubic centimetres. This quantity of salt solution is divided into 1000 parts, called thousandths.

The standard of an alloy of silver is generally the number of thousandths of solution of salt necessary to precipitate the silver contained in a gramme of the alloy.

*Measurement of the Solution of Common Salt.*—The solution of common salt will hereafter be termed the normal solution of common salt. It can be measured by weight or volume. The measure by weight gives greater precision, and it has the special advantage of being independent of temperature; but it requires too much time in numerous assays. The measure by volume gives a sufficient exactitude, and requires much less time than the measure by weight; it is, indeed, liable to the influence of temperature, but tables for correction will be appended.

Fig. 82.

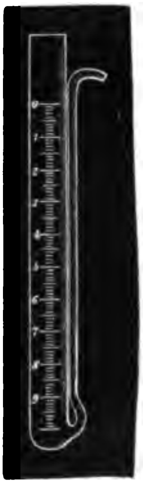


*Measure of the Normal Solution of Salt by Weight.*—This solution should be so made that 100 grammes will exactly precipitate 1 gramme of pure silver dissolved in nitric acid. In order to point out the method of taking the weight it must be supposed to have been previously prepared. After the process taking the weight is described, the mode of preparing the solution will be given.

The solution is weighed in a burette (fig. 82), whose capacity is from 115 to 120 grammes of the solution, and divided into grammes. These divisions are for the purpose of approximatively determining the weight of solution, so as to shorten the operation of weighing. The burette is represented as closed by a cork, *B*, in order to prevent evaporation of the solution when the instrument is not in use. It is also easy to remedy the inconvenience of evaporation, by rinsing the burette with a small quantity of the fresh solution. On pouring the solution from the orifice, *O*, of the burette, each division will furnish from 8 to 10 drops; and consequently the weight of a drop is about a decigramme. The burette is filled with solution to the division *o*; it is then tared in a balance capable of turning with a centigramme. The burette is then removed, and its place supplied with a weight equivalent to the amount of solution required—100 grammes, for instance. The solution is then gradually poured from the burette into a bottle appointed for its reception, until the equilibrium is nearly established. It is not easy to attain the point exactly, as no smaller quantity than a drop can be poured from the burette. This, however, is a matter of indifference; it suffices to know the exact weight of the solution poured out: suppose it to be 99 gr. 85 c.: the mode of more nearly approximating the required weight of 100 grammes will now be pointed out.

It must be remarked that it is not the amount of water contained in the 100 grammes that is of consequence, but only the quantity of salt found in solution; this should exactly represent 1000 thousandths of pure silver. If near 100 grammes of the normal solution be mixed with 900 grammes of water, it is evident that 1 gramme of this new solution is equivalent to a decigramme of the first, and consequently it will be easy to obtain 100 grammes of the normal solution, or rather the 1000 thousandths of salt it ought to contain: it will now be sufficient to add to the 99 grammes already poured from the burette, 1½ grammes of the new solution. It can be weighed, like the normal solution, to a drop nearly, in the burette (fig. 83), of such a diameter that each small division represents a decigramme of liquid, and consequently a centigramme of the normal solution; but it is more readily measured by volume, preparing it in the manner to be hereafter pointed out. To avoid all confusion, a solution to be termed a *decime* solution of common salt is one containing the same quantity of salt

FIG. 83.



as the normal solution, in a weight or volume ten times greater.

A *decime* solution of silver is a solution of silver equivalent to the latter, both mutually suffering complete decomposition.

*Preparation of the Decime Solution of Common Salt.*—One hundred grammes of the normal solution of common salt are weighed in a flask (fig. 84) containing a kilogramme of pure water, when filled up to the mark *a b*, or 1000 cubic centimetres; this quantity is made up with pure water, taking care to agitate the whole well, to render the mixture homogeneous. A cubic centimetre of this solution represents 1 thousandth of silver. This quantity is readily obtained by means of a pipette (fig. 85), gauged so that when filled up with water to the mark *c d*, it shall allow 1 gramme, or 1 cubic centimetre, to run freely, the small quantity of liquid remaining in the pipette not forming part of the gramme. In pouring the liquid by drops, a little more or a little less than twenty may be counted, according to the size of the orifice, *o*. This number will not vary more than one drop. Half a cubic centimetre will consequently be represented by 10 drops, and a quarter by 5. The precision arrived at by this method of measurement suffices, since the possible error on the cubic centimetre will be but one-twentieth of that quantity, or one-twentieth of a thousandth; if, however, many measures be required, then compensation must be made.

FIG. 84.

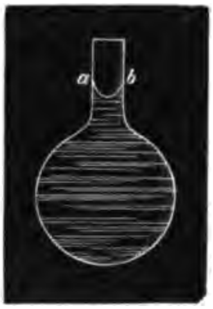
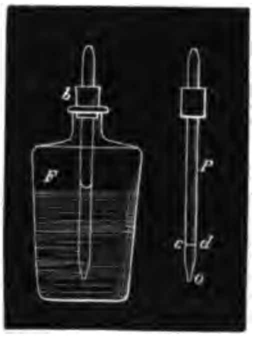
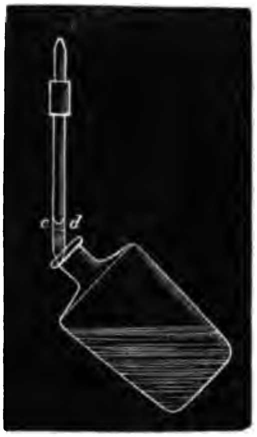


FIG. 85.



The decime solution of common salt requisite for assays must be kept in a bottle (fig. 85) closed by a cork, traversed by the pipette firmly fixed in a hole bored for that purpose. To measure a thousandth with the pipette, the bottle is held with one hand, and the pipette with the other (fig. 86). The pipette is taken from the solution after its upper orifice has been closed by the forefinger; the lower orifice is then inclined against the edge of the flask to remove the liquid, which without this precaution would remain there: the mark *c d* is then raised to the level of the eye, and by a suitable pressure of the forefinger on the upper orifice, which may be obtained by giving the pipette a slight alternating circular movement between the fingers, the solution is allowed to run out gradually. The instant the concave surface of the liquid is at the level *c d*, the pipette is firmly closed by pressure of the forefinger on its orifice, which is held above the bottle into which the solution is to be poured, and the forefinger removed so that it can be emptied. It is here necessary to remark, that in order to regulate the slow and regular runnings of the liquid from the pipette, by the pressure of the forefinger, the latter ought to be neither too moist nor too dry: if too dry it will not perfectly close the orifice, even by strong pressure; if too moist, it prevents the entrance of air, and the liquid will not run, or if it do, it will be irregularly. This observation should not be lost sight of in the use of the large burettes mentioned hereafter.

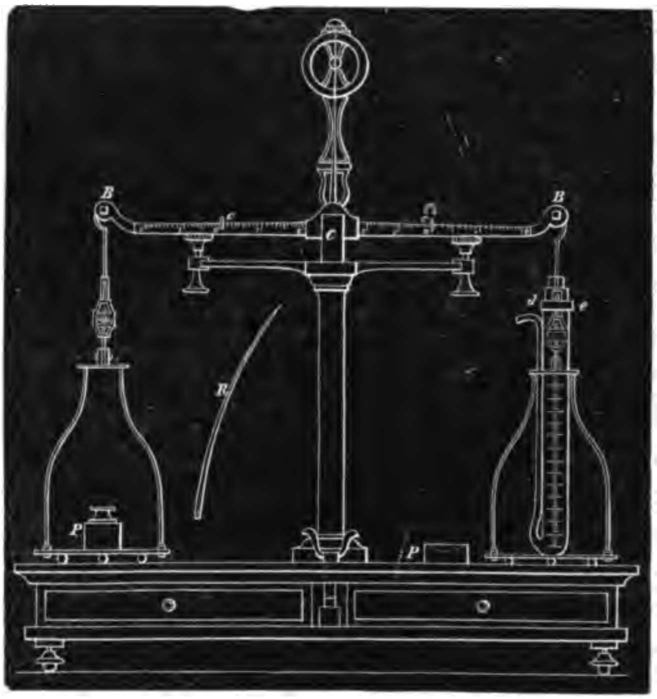
Fig. 86.



*Preparation of the Decime Solution of Silver.*—The decime solution of silver is prepared by dissolving 1 gramme of pure silver in nitric acid, in a flask holding 1 litre (see fig. 84), and then diluting the solution with distilled water so that, cooled at the ordinary temperature of the air, it shall occupy exactly the volume of one litre. It is measured in precisely the same manner as the decime salt solution.

*Weighing the Normal Solution of Common Salt.*—To execute this operation with rapidity, a balance similar to that represented at fig. 87 is employed. The arms are divided as in the assay balance described at p. 25; each of the arms, *C B* and *C B*, are furnished with a rider, *c*, of such a weight (about 5 decigrammes) that moved from the right or the left of the centre *o*, of each arm, it indicates two decigrammes. The space traversed by the rider is divided into twenty equal parts, representing an equal number of centigrammes.

Fig. 87.



We will take for example the weighing of 100 grammes of normal solution of common salt, which is that most frequently made in the determination of the standard of all varieties of argentiferous matter.

There are two weights, one, *P*, equal to the tare of the burette when full of solution to the mark *o*, the other, *P′*, equals 100 grammes. The burette is filled with solution, and placed on the right hand pan of the balance, on which it is kept in position by the collar *d e*, and through which it is passed before placing it on the pan. The tare, *P*, of the burette is supposed to be on the opposite side. If the equilibrium be not perfect, it is effected by the rider on the left; the burette is then removed, and 100 grammes of the solution (either more or less to one or two decigrammes) poured out. The burette is then again placed in the balance, with the 100 gramme weight *P′*, the upper part of which is slightly concave, to receive the bottom of the burette, in order to prevent it sliding off. The equilibrium is again established by the aid of the rider on the right. If, for instance, it is found necessary to remove the rider 15 divisions towards *B*, which represents 15 centigrammes, the weight of the solution poured out of the burette will be equal to 100 gr.—0·15 gr. = 99·85 gr. If, on the other hand, it is necessary to move the rider six divisions towards *C*, the weight of the solution will be 100 gr. + 0·06 gr. = 100·06 gr.

The above method of weighing the salt solution appears to be the most convenient that can be employed, although it is not very expeditious. Other methods of weighing and measuring will be given in an appendix to this article.

*Preparation of the Normal Solution of Common Salt when measured by weight.*—After having pointed out the method of weighing the normal solution of salt, and of taking very small quantities, its preparation will be described.

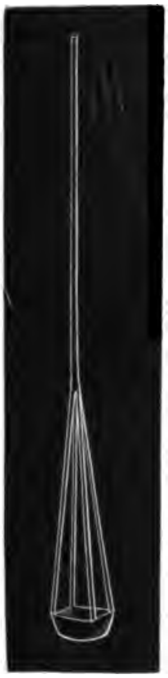
Supposing the salt as well as the water to be employed are pure, the two substances have only to be taken in the following proportions:—0·5427 kilogrammes of salt and 99·4573 kilogrammes of water, to form 100 kilogrammes of solution, of which 100 grammes will exactly precipitate 1 gramme of silver. But instead of pure salt which is difficult to procure, and which besides rapidly alters by the absorption of atmospheric moisture, it is preferable to employ a concentrated solution of commercial salt, which can be prepared in large quantities, and kept for use as needed. The quantity of salt it contains can be ascertained by evaporating a portion to dryness, and by a few experiments it is easy to determine in what proportion it shall be mixed with water to produce a solution, 100 grammes of which shall exactly precipitate 1 gramme of silver.

Suppose, for example, that the salt solution contains 250 grammes of salt per kilogramme, and that it is necessary to prepare 100 kilogrammes of the normal solution. Now, since for the preparation of this quantity 0·5427 kilogrammes of pure salt is required, we have the following proportion:—

$$0{\cdot}250 : 1 :: 0{\cdot}5427 : x = 2{\cdot}1708 \text{ kilogs.}$$

To this last weight enough water is added to make up 100 kilogrammes, that is to say, 97·8292 kilogrammes, which quantity can be readily measured by means of a flask containing 5 or 6 kilogrammes previously gauged.

FIG. 88.



The mixture must be well agitated by means of the agitator (fig. 88), which is made of an ozier twig, split into four branches, to the extremities of which is attached a small square piece of silk. This substance is employed to avoid the separation of filaments which would ensue from the use of any other material. This agitator can be introduced into very small openings, and is exceedingly serviceable in agitating large masses of liquid.

FIG. 89.

When well mixed, the solution must be assayed. To effect this, dissolve 1 gramme of silver in nitric acid, sp. gr. 1·290, in a stoppered bottle (fig. 89) holding about 200 grammes of water, tare the burette, fig. 82, filled with the solution, and pour rather more than less into the bottle; in proportion as the salt employed is impure, more than 100 grammes will be required to precipitate 1 gramme of silver. The mixture is at first milky, but, by vigorously shaking the bottle, having its stopper firmly fixed, for about a minute, and then allowing it to remain at rest for a short time, the liquid will become perfectly bright; two drops of the solution must then be poured into it from the burette: if a cloudiness is produced, it is agitated again to brighten it, and two drops more added. This must be continued until the last two drops added give no precipitate. The operation is then terminated, and nothing remains to state but the result.

Supposing the total weight of solution poured from the burette is 101·880 grammes, the last two drops must not be reckoned, because they produce no effect; the two preceding drops were necessary, but in part only; that is to say, that the number of drops to be deducted is less than four, and more than two, or rather that it is the mean term, three. Or the weight of a drop can be known exactly by taking that of a dozen: suppose it is equal to 0·082 gramme, three times that number must be deducted, or 10·255 grammes from 101·880 grammes: there will remain 101·625 grammes, representing the quantity of normal solution necessary to precipitate 1 gramme of silver.

The solution is thus found to be too weak; to bring it to its proper standard it is necessary to remove 1·625 grammes of water from the 101·625 grammes of solution, or, what is the same thing, to add to the normal solution a certain quantity of the concentrated solution of common salt, which quantity may be found by the following proportion:—

100 : 1·625 : : 2·1708 kilogrs. of silver solution : $x = 0{\cdot}0353$.

After the addition of this quantity of salt to the normal solution, a fresh assay is made, proceeding in precisely the same manner as before; taking care, however, to pour from the burette a weight of solution slightly under 100 grammes, or 1000 decigrammes; for instance, 998·4 decigrammes, because it is not possible, in pouring the solution by drops, to arrive at the exact weight, 1000 decigrammes. To ascertain the true standard in the most exact manner possible, a decime solution must be prepared by weighing 100 grammes of the normal solution, and diluting it with pure water, so that it shall occupy one litre: a cubic centimetre of this solution will represent a decigramme of the normal solution. This decime solution will not be rigorously exact, since the normal solution has not been truly standardised; but it is easily perceived that the error thus committed is very small, and that it may be neglected. Nevertheless, as soon as the normal solution is perfectly standardised, it is better to prepare another decime solution.

A decime solution may be immediately obtained by dissolving 0·5427 gramme of pure sea salt in such a quantity of water that the whole will occupy one litre; yet the first process is preferable.

With the decime solution the assay may be thus continued, remembering that the pipette described at fig. 83 is a cubic centimetre containing 20 drops; that the half therefore is represented by 10 drops, and the fourth by 5.

To the 998·4 decigrammes of normal solution already added, pour one pipette and 12 drops of the decime solution, which will exactly complete the weight of 1000 decigrammes of normal solution. The mixture is agitated to brighten it, and one-thousandth of common salt or one pipette of the decime solution added. If this causes a cloudiness, it is agitated and a second thousandth added. This last should produce no opalescence. The weight of normal solution necessary to exactly precipitate one gramme of silver will be between 1000 and 1001 decigrammes; that is to say, the mean will be equal to 1000½. The standard of the normal solution is then too weak by half a thousandth; to correct this a quantity of concentrated salt solution must be added equal to half a thousandth of that already added (2·1708 + 0·0353 = 2·2061 kilogrammes); that is to say, 1·1 gramme.

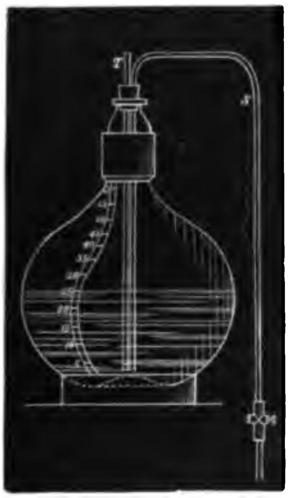
A new assay is then made for verification.

When the standard of a solution is very nearly arrived at, it is well to employ filtration to detect the slightest opalescence, at least when sufficient time is not allowed for the liquid to become perfectly bright. The surest method, when the standard is nearly attained, is to place some of the liquid in two test glasses, and pour into one a few drops of the decime solution of common salt, and into the other a corresponding number of drops of the decime solution of nitrate of silver. It may then be determined on which side the opalescence is manifested, and the assay of the normal solution may be continued after the mixture of the liquids in the two glasses, since the two quantities of the decime solutions of common salt and nitrate of silver mutually decompose each other, and do not interfere with the assay. Once the standard of the normal solution being definitely fixed, the sum of the quantities of the concentrated solution of common salt which have been employed, as well as those of the water, must be noted, and in the preparation of a new normal solution the proportions found as above would only have to be mixed to obtain at once a solution having very nearly its true standard.

In determining the standard of the normal solution, suppose that it were always too weak, it would be necessary to add to the solution a certain quantity of common salt; but if the true amount had been exceeded, and it had been found too strong, the solution would have to be precipitated with the decime solution of silver; and knowing the number of cubic centimetres or thousandths of silver which had been necessary to precipitate the excess of common salt, it could be determined what amount of water must be added to reduce the normal solution to standard. For instance, if 2 thousandths of the decime solution of silver had been consumed, 2 thousandths of its weight of water would have to be added to the total amount of solution; that is to say, 0·2 kilogramme or 200 grammes.

*Preservation of the Normal Solution of Common Salt.*—The most suitable vessel for containing the normal solution of common salt is one of glass, because that cannot affect the standard. Large black glass bottles, termed *carboys*, are found in commerce. These bottles contain from 50 to 60 litres, and are very applicable for this purpose. Fig. 90 represents one of these bottles fixed in a stand formed of a sieve hoop. It is graduated into litres or kilogrammes of water, and a paper scale fixed on its side shows at any time the quantity of contained liquid. It is closed by an hydraulic valve, made of sheet-iron, but the bell or cover is of glass. The detail of this valve is shown at fig. 91. The air can only enter the bottle by the narrow tube *T*, and cannot pass out by it; consequently, evaporation is not to be feared. The neck of the valve should be about a decimetre deep, into which mercury should be poured, but only to about one-third of its height.

Fig. 90.



The solution is drawn from the bottle by the syphon *S*. This is furnished with a stopcock; but this syphon being brittle, at least when not of metal, is not convenient in use, since it is incorporated with the bell of the valve: it is, therefore, preferable to pierce the bottom of the bottle (fig. 92), and fix a metal tube (*T*) by means of a plate moulded on the bottom, and cemented to it. This tube is raised a little above the bottom of the bottle, and covered by a small hoop, the object of which is to protect it from any of the mercury which might fall into it. It is terminated at its other extremity by a very narrow tube, so that the flow of

Fig. 91.

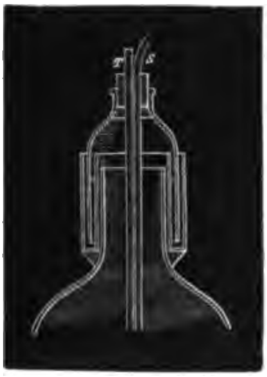
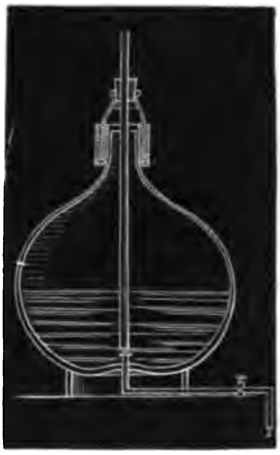


Fig. 92.



the solution may not be too rapid. Hereafter a metal reservoir will be described which has all the advantages of a glass vessel without its inconveniences.

*Application of the Process described in the Determination of the Standard of a Silver Alloy.*—The alloy is supposed to be that made into coin, the mean standard of which is fixed at 900 thousandths, but which may vary from 897 to 903 thousandths without ceasing to be legal (French standard for coin). One gramme is dissolved in the bottle (fig. 89) by about 10 grammes of nitric acid, sp. gr. 1·290. This quantity of nitric acid can be readily taken by means of the pipette *P* (fig. 93), which contains 7·7 grammes of water to the mark *a b*. The solution may be accelerated by placing the bottle in a small saucepan of hot water, the bottom of which must be covered with a piece of cloth, so as to prevent contact of the glass and metal. The solution finished, and the flask slightly cooled, the nitrous vapour must be removed by a blower (see fig. 94), the nozzle of which is formed of a piece of bent glass tube, connected by a cork with a copper socket *D*, having a screw inside. This operation ought to be effected, as well as the solution of the alloy in nitric acid, under a chimney with a strong current of air, to carry off the nitrous vapour.

FIG. 93.

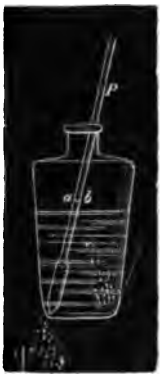
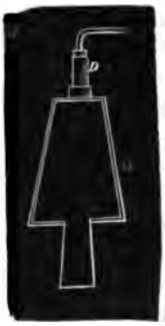


FIG. 94.



The burette (fig. 82), being filled with the normal solution of common salt, and tared, about 90 grammes are poured into the solution of the alloy; say 89·85 grammes. After agitating the liquor, a cubic centimetre of the decime solution of common salt is added, representing one thousandth of silver. If a cloudiness be observed, agitate again, and add a second thousandth of common salt, and so on, until the last thousandth gives no precipitate. Suppose it to be the fourth: that must not be counted, because it has produced no effect; and only half of the third must be taken, because only a portion of that was necessary. The standard of the alloy would be consequently equal to nearly half a thousandth, to 898·5 + 2·5 = 901.

If it be desirable to approach still nearer to the true standard of the alloy, half-thousandths must be added until the last half thousandth gives no precipitate; and in order to avoid all confusion, it is better to write with chalk on a black-board the thousandths of common salt, preceding them by the plus sign +, and on the other side the thousandths of nitrate of silver, preceding them by the sign − minus.

In the above example, after the addition of the 4 thousandths of common salt, the last of which has produced no cloudiness, 1½ thousandths of nitrate of silver are added, which destroy 1½ thousandths of common salt, and brighten the liquid. If another half thousandth of nitrate of silver produce no precipitate, it is not taken into account, and is struck off from the table. From whence is concluded that the quantity of nitrate of silver necessary to destroy the excess of common salt is more than 1 and less than 1½; that is to say, nearly the ¼ of a thousandth, and is equal to 1¼. Thus the number of thousandths of salt really used is 4 − 1·25 = 2·75. The standard of the alloy, therefore, is 898·50 + 2·75 = 901·25.

Another example, everything else remaining as above; but the first thousandth of salt did not precipitate. This is a proof that too much normal solution of common salt has been employed, and that there is an excess of salt in the liquid. Add one thousandth of silver, and agitate: things are now as at first, but it is nevertheless known that it is with nitrate of silver the process must be continued. One thousandth has been added, which produced a precipitate; the second does not. The standard of the alloy is consequently 898·50 − 0·5 = 898. To approach still nearer to the real standard, destroy the last two thousandths of silver by two thousandths of common salt, and add half a thousandth of silver—a cloudiness is produced, as already known; but another half thousandth does not precipitate. The standard of the alloy is therefore 898·50 − 0·25 = 898·25.

This process, on which it would be useless to enlarge further at present, because many other parts of the process to be presently described apply to it, is general, and gives exactly the standard of an alloy when it is known approximatively, which can always be ascertained by a previous rough assay.

### ASSAY BY THE HUMID METHOD, MEASURING THE NORMAL SOLUTION OF COMMON SALT BY VOLUME.

The measurement by weight of the normal solution of common salt has, as already stated, the advantage of being independent of temperature, of having the same degree of precision as the balance, and of requiring no correction. The measurement by volume has not all these advantages; but, by ensuring an adequate amount of accuracy, it has that of being more rapid, and renders the new process applicable to numerous and daily assays.

The normal solution of common salt measured by volume is so prepared that it has a volume equal to that of 100 grammes of water, or 100 cubic centimetres, and at a determinate temperature exactly precipitates 1 gramme of silver. The solution can be kept at a constant temperature, in which case the assay requires no correction; or, if the temperature be variable, its influence on the assay must be corrected. These two circumstances do not change the principle of the process; but they are sufficiently important to require some changes in the apparatus, and that each of the two processes should be treated separately: one, in which the normal temperature is constantly maintained; the other, in which it is variable. Experience has shown the latter to be preferable, and it will be first detailed; the other will be described hereafter.

*Methods of Measurement in the employment of Volumes instead of Weights.*—It will be here admitted, in pointing out the methods of measuring the normal solution of common salt by volume, that it has been already prepared, and even, that it is kept at a constant temperature. It will afterwards be very easy to describe the method of preparation, and give the corrections of which it is susceptible when its temperature varies.

A volume of solution of 100 cubic centimetres is readily obtained by means of a pipette (fig. 95), graduated so that, filled with water to the mark *a b*, and the point or jet well wiped, it will allow 100 grammes of water, at a temperature of 15° (centigrade), to flow in a continuous stream. A continuous stream is expressly mentioned, because sometimes after the cessation of the jet the pipette will yet give two or three drops of liquid, which must not be counted. The weight of the volume of normal solution taken in this manner with suitable precautions will be constant, from one extreme to another, to 2½ centigrammes, or rather to ¼th of a thousandth.

Fig. 95.

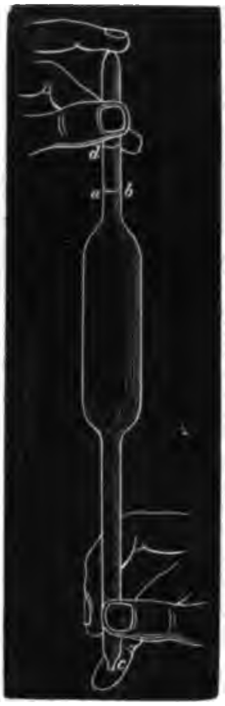
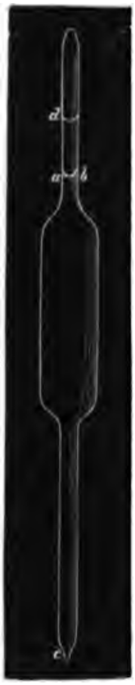


Fig. 96.



The following is the most simple method of taking a measure of the normal solution of salt:—

Immerse the jet (*c*) of the pipette in the solution, apply the mouth to the upper orifice, and draw the liquid into *d*, above the circular mark *a b*. Dexterously apply the forefinger of one of the hands to this orifice, remove the pipette from the liquid, and hold it as represented at fig. 95. The mark *a b* is held on a level with the eye, and the surface of the solution allowed to descend until it forms a tangent with the plane *a b*. At this instant the jet (*c*) of the pipette is set at liberty by removing the finger against which it had been pressed, and, without otherwise changing the position of the hands, the contents are allowed to run into the bottle appropriated for that purpose, taking care to remove the pipette as soon as the steam stops.

If, after having filled the pipette by aspiration, there is any difficulty found in a sufficiently rapid application of the forefinger to the superior orifice to prevent the fall of the liquid below the mark *a b*, the pipette must be removed from the liquid, the orifice being closed by pressing the tongue against it: then apply the middle finger of one of the hands to the lower orifice, remove the tongue, and apply the forefinger of the other hand to the larger orifice, previously wiped dry.

Fig. 97.

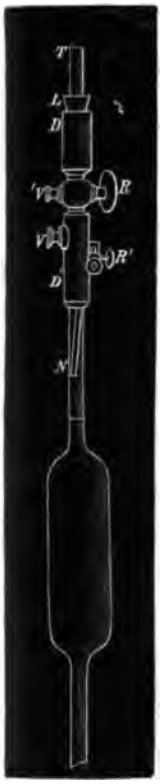


Fig. 98.

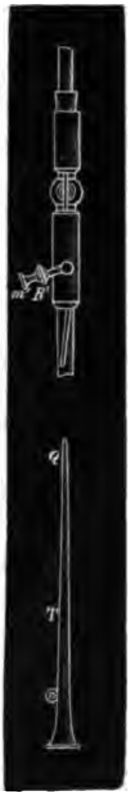
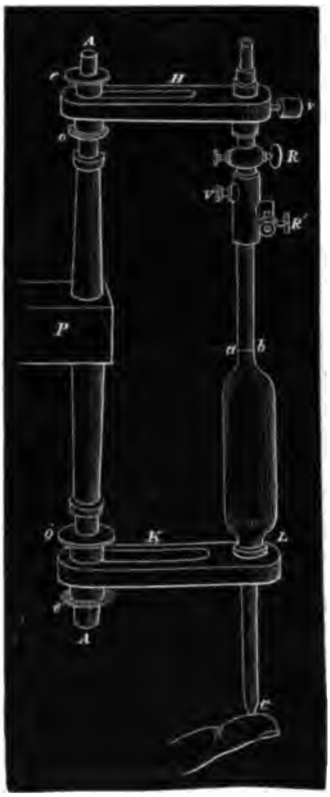


Fig. 99.



The process just described for obtaining a measure of normal solution of salt is exceedingly simple, because it requires but little apparatus; but another, of more easy execution, will now be mentioned, and which is at the same time more exact.

In this process the pipette is filled from above, like a bottle, instead of by aspiration; furthermore, it is a fixed apparatus. The figure 97 represents this apparatus. *D D′* are two sockets, separated by a stopcock *R*. The upper one, which is screwed inside, is connected by means of a cork, *L*, with the tube *T*, which conducts the solution of salt. The lower socket is cemented to the pipette; it is furnished with an air-tap *R′*, and a screw *V*, which serves to regulate the admission of air into the pipette by a small opening provided for that purpose. Below the stopcock *R′*, and soldered to the socket, is a very narrow silver tube *N*, conducting the solution into the pipette, and allowing the escape of displaced air by the air-tap *R′*. The thumbscrews *V′* replace the ordinary screw, by means of which the key of the cock is adjusted on its seat.

The figure 98 represents the above described apparatus on the other side. There will in this be noticed on the air-cock *R′*, an opening *m*, into which is ground by its extremity *Q* the conical tube *T* (same figure). By this, air can be drawn out of the pipette whenever it is desirable to fill it from below.

The pipette is carried by two horizontal arms, *H K*, fig. 99. These arms are movable around a common axis *A A*, and are also capable of moving in the two longitudinal slots.

They are fixed by two nuts, *e e′*, and their distance can be changed by means of pieces of wood or cork interposed, or even by the other nuts, *o o′*. In the upper arm, *H*, is a hole, in which is fixed by a wooden thumb-screw, *v*, the socket of the pipette; the corresponding hole of the lower arm is larger; the jet of the pipette is kept in position by a cork, *L*. The apparatus is fixed by its appendage, *P*, by means of a screw on an angle of the wall, or any other support.

The method of filling this pipette is very simple: apply the forefinger of the left hand to the orifice, *c*, then open the two stopcocks, *R*, and *R′*; when the liquid nears the neck of the pipette its flow is moderated, and as soon as it is a little above the mark *a b*, the stopcocks are shut, and the forefinger removed. The pipette must now be accurately adjusted, so that the liquid touches the mark *a b*, and none remains on the outside of the jet *c*.

This last condition is easily fulfilled: after having removed the finger by which the orifice *c* of the pipette was closed, a moist sponge, *m*, fig. 100, enveloped in linen, is then applied, which absorbs the excess of liquid. To abridge the description, this sponge will be termed 'the handkerchief,' and the pipette is said to be clean when no liquid adheres exteriorly to the orifice.

For convenience in use the handkerchief is forced into a tube of tin-plate, terminated by a little cup, open below, so that the liquid may run into the vessel *C*, on which the tube is soldered: the liquid from the handkerchief is rejected; it can be easily removed to wash it, and if necessary it can be pushed towards the pipette by a small wedge of wood, *o*.

Fig. 100.

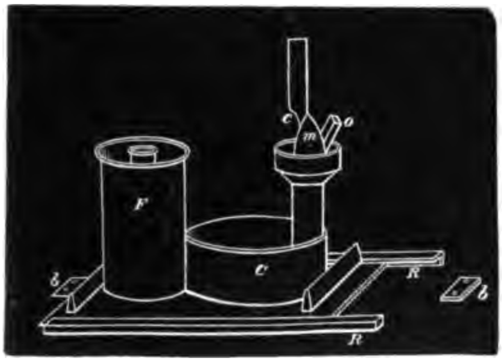
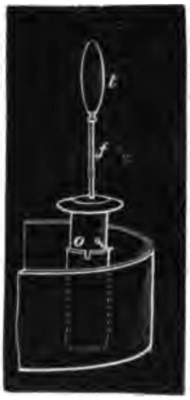


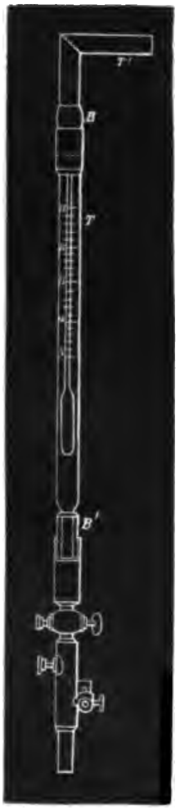
Fig. 101.



At a later period the following mode of making the handkerchief has been found preferable: on a double iron wire (fig. 91) forming a spring, a small band of tin-plate *t*, is rolled; the iron wire is cemented into a tin-plate cylinder, closed on the lower end, and furnished on the upper with a border to convey the liquid which runs from the handkerchief in the vessel *C*. This cylinder passes into another soldered to the bottom of the vessel, and can be kept in position by two projections *o*, which work in two slots cut in the other cylinder.

To complete the adjustment of the pipette, the liquid must be made to fall to the level *a b*. To this end whilst the handkerchief is in contact with the jet of the pipette, air is allowed to enter slowly by unscrewing the screw *v* (fig. 99), and the instant the level is attained the handkerchief is removed, and the bottle *F* (fig. 100), which is employed to receive the solution, placed under the jet of the pipette. This must be accomplished rapidly, and without hesitation. The bottle is then placed in a cylinder of tin-plate, whose diameter is just a little larger, and which forms part and parcel of the vessel *C*, and the handkerchief. The whole of this apparatus has a sheet of tin-plate for a base movable between two wooden rods, *R R*, each having a slot in which the tin-plate moves. The extent of its movements is determined by two pieces of wood, *b b*, so placed that when it is stopped by them, the jet of the pipette corresponds to the centre of the neck of the bottle, or by the other in contact with the handkerchief. This arrangement is exceedingly handy for wiping and emptying the pipette, and has a sufficient amount of solidity to allow of its being removed and replaced without injury. It will be readily seen that when the admission of air into the pipette has been once regulated by the screw *v*, it will be advantageous to leave it so, because the movement of the handkerchief or bottle can be so rapidly effected, that a drop of the liquid has not time to accumulate and fall.

FIG. 102.



*Temperature of the Solution.*—Having described the method of measuring the volume of the normal solution of salt, that which appears the most suitable of obtaining the temperature will be pointed out.

The thermometer is placed in a glass tube, *T* (fig. 102), through which the solution passes, running into the pipette. It is suspended by a cork having four channels cut in it to allow the free passage of the liquid. The scale is engraved on the tube itself, and is repeated on the opposite side, so as to fix the eye by this double scale to the height of the thermometric column. The tube is fused at its lower end to a narrower tube, which is fixed by means of a cork into the socket of the stopcock of the pipette. The upper part of this tube is cemented to a socket of copper, tapped inside, which in its turn is fastened by a cock *B*, with the extremity (also tapped) of the tube *T'*, communicating with the reservoir of normal solution. The corks used as joints between the parts of the apparatus retain a certain amount of flexibility, and allow it being taken to pieces and put together again in a short space of time; but it is essential to pass them into a hollow tube of glass or metal, to prevent them giving way under the pressure they have to sustain. If care be taken to coat them with a little tallow to stop the pores, no escape need be apprehended.

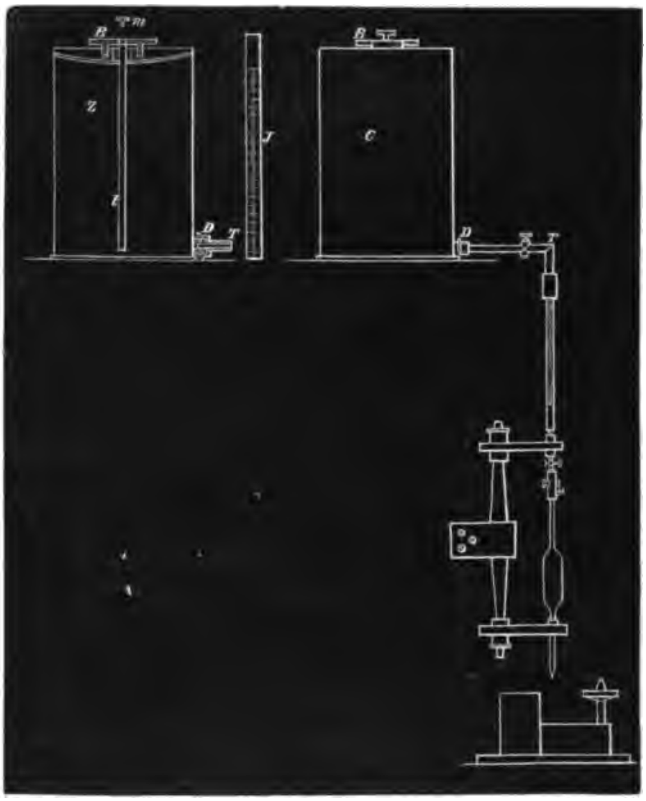
*Preservation of the Normal Solution of Salt in Metallic Vessels.*—This subject has already been discussed, and it may appear unnecessary to again refer to it; but as it is here a question of metallic vessels, some details seem necessary.

The figure 103 represents a cylindrical copper vessel, *C*, holding about 110 litres. It is seen in section, *Z*, same figure. To its base is soldered a socket, *D*, to which is adapted a tube, with stopcock, *T*, through which the solution passes into the pipette; the upper part, which is slightly concave, having an opening closed by a screw stopper, *B*, the edges of which press on a washer. This stopper is traversed by the tube *t*, which passes nearly to the bottom of the vessel, and through which air enters the apparatus, without the power of again passing out, so that evaporation is effectually prevented. This tube can be closed by a stopper, *m*, when the apparatus is not in use.

The quantity of liquid contained in the vessel can be determined at any time by the aid of a wooden gauge, *J*, graduated into litres. When used it is plunged vertically into the liquid, but is seldom needed.

Pure or tinned copper alters in contact with the solution of salt and air, and the solution continually decreases in strength. This inconvenience is remedied by coating the inside of the cylinder with a soft cement, such as described at page 108; or with that cement softened by the addition of one-third its weight of yellow wax. This operation may be performed

FIG. 103.



by removing the tubes *T* and *t*, perfectly cleansing the inside of the cylinder, and heating it. About four or five pounds of the cement, made very hot, are run in, and the cylinder so turned round and inverted that the cement may run over every part. The turning is continued until the cement is cold. All the parts just described are united in the figure 103, forming a complete apparatus for the preservation of the normal solution of salt, for observing the temperature, and for measuring the volume.

*Preparation of the Normal Solution of Salt, measuring by Volume.*—The preparation of the normal solution of salt, measured by volume, is much the same as of the solution measured by weight; there is, consequently, very little to add to that already given at pages 501–505, and to which the reader is referred.

The cylinder, as already supposed, will contain about 110 kilogrammes of water: no more, however, than 105 are put in; so that sufficient space may remain in order to agitate the fluid without throwing any out. According to the condition imposed, that 100 cubic centimetres, or one-tenth of a litre, of solution, should contain sufficient salt to completely precipitate 1 gramme of pure silver; and further, admitting 13·516 for the equivalent of silver, and 7·335 for that of salt, the quantity of pure salt to be dissolved in 105 litres of water, and which corresponds to $105 \times 10 = 1050$ grammes of silver, will be found by the following equation:—

$$13{\cdot}516 : 7{\cdot}335 :: 1050 \text{ gram.} : x = 569{\cdot}83 \text{ gram.}$$

And as the solution of commercial salt employed, page 501, contains approximatively 250 grammes per kilogramme, 2279·3 grammes of this solution will be required to furnish 569·83 grammes of salt. As the 2279·3 grammes of solution contain 569·83 grammes of salt, it will consequently contain 1709·5 grammes of water, which must be taken into account in measuring the 105 litres: that is no more than about 103·3 must be employed. The whole being well mixed, the tubes and pipette must be washed out several times, by allowing the solution to run through them. The solution so passed is again placed in the cylinder, and after each addition the contents are well agitated, and lastly, the standard of the solution is determined, the temperature being supposed to remain constant.

To accomplish this more readily, two decime solutions are prepared; one of silver, and the other of salt.

The decime solution of silver, as already stated, is obtained by dissolving a gramme of silver in nitric acid, and diluting the solution with water until its volume is one litre.

The decime solution of salt can be obtained by dissolving 0·543 grammes of pure salt in water, so that the solution fills a measure of one litre; but it is best prepared with the normal solution itself, which is to be standardised, by mixing one measure of the latter with nine measures of water. It must, however, be understood, that this solution is not rigorously equivalent to that of the silver, and only becomes so when the normal solution employed in its preparation becomes fixed at its true standard. If the normal solution be correct to ten thousandths, or one hundredth, the decime solution may be correct to the same degree. If ten thousandths of the latter solution be employed, the error committed will be one-tenth of a thousandth; and only one hundredth when one thousandth is employed. Such errors may be entirely neglected; nevertheless, after having exactly standardised the normal solution, it is better to prepare a new decime solution.

After the preparation of the decime solutions, several bottles, as at fig. 89, must be prepared, each of which contains 1 gramme of pure silver dissolved in 8 or 10 grammes of nitric acid. To these will be given the name of check, or witness-assays.

To ascertain the standard of the normal solution pour a pipetteful into one of the check flasks, and agitate briskly until quite bright. After a few moments' repose, two thousandths of the decime solution of salt are added, which, by superposition, will produce a precipitate. The normal solution is consequently too weak, since the salt employed was not perfectly pure. It is again agitated, and two other thousandths are added, which produce a precipitate. The addition of successive two thousandths is thus continued until the last produce no precipitate. Suppose in all sixteen thousandths have been added: the two last which have been added are not reckoned, as they produce no precipitate: the two preceding have only been in part necessary; that is to say, that the acting thousandths added are above 12 and below 14, or, taking the mean, equal to 13.

Thus in the existing state of the normal solution 1013 parts are necessary to precipitate 1 gramme of silver, while only 1000 should be required. The quantity of concentrated solution of common salt to be added may be found by noting that the quantity of solution of common salt first employed—that is to say, 2279·3 grammes—has only produced a standard of $1000-13=987$ thousandths, and by the following equation:—

$$987 : 2279{\cdot}3 :: 13 : x = 30{\cdot}02 \text{ grammes.}$$

This quantity of solution of common salt must, therefore, be mixed with the normal solution.

After having washed the tubes and pipette with a new solution, another check gramme of silver is operated on. It is found, for instance, by proceeding but by one thousandths at a time, that the first precipitates, but the second does not. The standard of the solution is therefore too weak, being comprised between 1000 and 1001; that is to say, it is equal to 1000½: this, however, is not sufficiently near.

Pour into the assay flask two thousandths of the decime solution of silver: these will merely decompose the two thousandths of salt, and the operation will have retrograded by two thousandths; that is, it will be reduced to the point from which the thousandths were first employed. If, after brightening the liquor, half a thousandth of the decime solution is added, there will necessarily be a precipitate, as was before known; but a second half thousandth produces no cloudiness. The standard of the normal liquid is therefore between 1000 and 1000½, or equal to 1000¼.

This for most purposes may be considered sufficiently near; but if it be desirable to correct it, it may be remembered that the two quantities of solution of common salt added,

$$2279{\cdot}3 \text{ gr.} + 30{\cdot}02 \text{ gr.} = 2309{\cdot}32 \text{ gr.}$$

have only produced 999·75 thousandths, and that it is necessary to add a fresh quantity corresponding to the quarter of a thousandth. The proportion is thus found:—

$$999{\cdot}75 : 2309{\cdot}32 :: 0{\cdot}25 : x.$$

But as the first term only slightly differs from 1000, it is necessary, in order to have $x$, to take $\frac{0{\cdot}25}{1000}$ of 2309·32, and 0·577 gr. will be found the quantity of solution of common salt to be added to the normal solution. It is not convenient to exactly take so small a quantity of solution of common salt by means of the balance, but is more readily attained in the following manner:—

Weigh 50 grammes of the solution, and dilute with water until it occupies exactly half a litre, or 500 cubic centimetres. A pipette of this solution, containing a cubic centimetre, will give a decigramme of the original solution; and as the pipette is divided into 20 drops, each drop will represent 5 milligrammes of the solution. Still smaller quantities may be determined by still further dilution, but greater precision is useless.

The standardising of the normal solution is much less tedious than may be supposed; and it must be remarked, that the liquid for a thousand assays is prepared at once, and moreover, that in preparing a fresh solution, its true standard may be very nearly obtained at once, if the quantities of water and salt solution previously employed have been noted.

*Correction of the Standard of the Normal Solution of Salt when the Temperature varies.*—It has been admitted that, in the determination of the standard of the normal solution of salt, the temperature has remained constant. Assays made under these circumstances need no correction; but if the temperature changes, the same measure of solution will not contain the same amount of salt. Supposing the solution of salt has been standardised at 15°. If, at the time an experiment is made, the temperature is 18°, for instance, the solution will be found too weak, since it has become expanded, and the pipette holds less than its weight. If, on the other hand, the temperature falls to 12°, the solution becomes concentrated, and is found too strong. It is therefore necessary to determine the correction to be made for any variation of temperature that may occur.

To this end the temperature of a solution of common salt has been gradually raised from 0....5....10....15....20....25....30 degrees, and three pipettefuls of the solution exactly weighed at each of the above temperatures. One-third of the total weight gives the mean weight of the contents of a pipette. The corresponding weights of a pipetteful of solution are then entered, and form the second column of the following table, called 'Table of Correction for the Variations of Temperature in the Normal Solution of Salt.' By this table correction may be made for any temperature between 0 and 30 degrees, when the solution of salt has been standardised within the same limits. Suppose, for example, the solution had been standardised at 15°, and that at the time it was used its temperature was 18°. On referring to the second column of the table, it will be seen that the weight of a measure of solution at 15° is 100·099 gr.; and at 18° 100·065 gr.; the difference 0·034 gr. is the quantity of solution taken too little, and consequently it must be added to the normal measure, so that it may be equal to one thousand thousandths. If the temperature of the solution had fallen to 10°, the difference of weight between a measure at 10° and a measure at 15° will be 0·019 gr., which must, on the contrary, be deducted from the measure, as it has been taken in excess. These differences of weight of a measure of solution at 15° and that of a measure for any other temperature, forms the column 15° in the table, where they are expressed in thousandths. They are written on the same horizontal line as the temperatures to which each corresponds, with the sign + when they are to be added, and the sign - when to be subtracted. The columns 5°, 10°, 20°, 25°, 30° have been calculated in the same manner, to meet cases in which the normal solution had been graduated at each of the above-named temperatures. Thus, to calculate the column 10°, take the number 100·118 from the column of weights as a point of departure, and find the difference for all the other numbers in the same column.

An application of this table will be given hereafter.

### Table of Correction for Variations in Temperature of the Normal Salt Solution.

| Temperature | Weight | 5° | 10° | 15° | 20° | 25° | 30° |
|---|---|---|---|---|---|---|---|
| Degrees | Grammes | Mill. | Mill. | Mill. | Mill. | Mill. | Mill. |
| 4 | 100·109 | 0·0 | —0·1 | +0·1 | +0·7 | +1·7 | +2·7 |
| 5 | 100·113 | 0·0 | —0·1 | +0·1 | +0·7 | +1·7 | +2·8 |
| 6 | 100·115 | 0·0 | 0·0 | +0·2 | +0·8 | +1·7 | +2·8 |
| 7 | 100·118 | +0·1 | 0·0 | +0·2 | +0·8 | +1·7 | +2·8 |
| 8 | 100·120 | +0·1 | 0·0 | +0·2 | +0·8 | +1·8 | +2·8 |
| 9 | 100·120 | +0·1 | 0·0 | +0·2 | +0·8 | +1·8 | +2·8 |
| 10 | 100·118 | +0·1 | 0·0 | +0·2 | +0·8 | +1·7 | +2·8 |
| 11 | 100·116 | 0·0 | 0·0 | +0·2 | +0·8 | +1·7 | +2·8 |
| 12 | 100·114 | 0·0 | 0·0 | +0·2 | +0·8 | +1·7 | +2·8 |
| 13 | 100·110 | 0·0 | —0·1 | +0·1 | +0·7 | +1·7 | +2·7 |
| 14 | 100·106 | —0·1 | —0·1 | +0·1 | +0·7 | +1·6 | +2·7 |
| 15 | 100·099 | —0·1 | —0·2 | 0·0 | +0·6 | +1·6 | +2·6 |
| 16 | 100·090 | —0·2 | —0·3 | —0·1 | +0·5 | +1·5 | +2·5 |
| 17 | 100·078 | —0·4 | —0·4 | —0·2 | +0·4 | +1·3 | +2·4 |
| 18 | 100·065 | —0·5 | —0·5 | —0·3 | +0·3 | +1·2 | +2·3 |
| 19 | 100·053 | —0·6 | —0·7 | —0·5 | +0·1 | +1·1 | +2·2 |
| 20 | 100·039 | —0·7 | —0·8 | —0·6 | 0·0 | +1·0 | +2·0 |
| 21 | 100·021 | —0·9 | —1·0 | —0·8 | —0·2 | +0·8 | +1·9 |
| 22 | 100·001 | —1·1 | —1·2 | —1·0 | —0·4 | +0·6 | +1·7 |
| 23 | 99·983 | —1·3 | —1·4 | —1·2 | —0·6 | +0·4 | +1·5 |
| 24 | 99·964 | —1·5 | —1·5 | —1·4 | —0·8 | +0·2 | +1·3 |
| 25 | 99·944 | —1·7 | —1·7 | —1·6 | —1·0 | 0·0 | +1·1 |
| 26 | 99·924 | —1·9 | —1·9 | —1·8 | —1·2 | —0·2 | +0·9 |
| 27 | 99·902 | —2·1 | —2·2 | —2·0 | —1·4 | —0·4 | +0·7 |
| 28 | 99·879 | —2·3 | —2·4 | —2·2 | —1·6 | —0·7 | +0·4 |
| 29 | 99·858 | —2.6 | —2·6 | —2·4 | —1·8 | —0·9 | +0·2 |
| 30 | 99·836 | —2·8 | —2·8 | —2 6 | —2·0 | —1·1 | 0·0 |

### TABLE FOR THE ASSAY, BY THE WET METHOD, OF AN ALLOY CONTAINING ANY PROPORTIONS WHATEVER OF SILVER, BY THE EMPLOYMENT OF A CONSTANT MEASURE OF THE NORMAL SOLUTION OF COMMON SALT.

The process by which the normal solution of salt is measured by weight is applicable to the assay of every kind of alloy, since it suffices to take a weight of the solution corresponding to the presumed standard of the silver, and complete the assay by means of the decime solution; the process by volume, however, has not the same advantage, because the volume of normal solution cannot be varied in the same manner as the weight. This inconvenience, however, is of no very great consequence, for, by keeping the volume of normal solution constant, it suffices to vary the weight of the alloy, taking in each particular case a weight which contains approximatively one gramme of pure silver. Suppose the alloy has a standard of about 900 thousandths, we have the following proportion:—

$$900 \text{ thousandths} : 1000 \text{ of alloy} :: 1000 \text{ thousandths} : x = 1111{\cdot}1.$$

If that weight be now taken to ascertain the standard of the alloy, it may be found, for instance, that to the measure of 1000 thousandths of salt it is yet necessary to add 4 thousandths of salt to precipitate the whole of the silver; that is to say, that 1111·1 of alloy really contain 1004 of silver. From this result the real standard of the alloy may be found to be 903·6, by the following equation:—

$$1111{\cdot}1 : 1004 :: 1000 : x = 903{\cdot}6$$

But such calculations, however simple, should be avoided where numerous daily assays are made, not only on account of the time consumed, but still more from the errors to which such operations are necessarily exposed. Fortunately, all these inconveniences may be avoided by the use of tables, which entirely free the assayer from calculation.

Wishing in weighing the alloy to avoid fractions of thousandths, and only making use of tenths and half-tenths of thousandths, the weight of alloy increases, starting from a gramme, from 5 to 5 thousandths, and the corresponding standard for each of these weights has been sought, all containing one gramme of pure silver. Thus the weight 1020 of alloy, in which there are 1000 of silver and 20 of copper, corresponds to the standard 980·39, obtained by the proportion—

$$1020 : 1000 :: 1000 : x = 980{\cdot}39$$

On this principle are formed the first and second columns of the table marked Salt. The first contains the weight of each alloy, and the second its corresponding standard. The following columns, 1, 2, 3, to 10, give the standard of the alloy, when, instead of the 1000 milligrammes of silver it was supposed to contain, it really contained 1, 2, 3, &c. more, and consequently 1, 2, 3, &c. milligrammes of copper less.

Another table, constructed in the same manner as the preceding, and marked Nitrate of Silver, gives the standard of the alloy when, under the weight given in the first column, it contains 1, 2, 3, &c. milligrammes less silver, and as much more copper. Thus, for example, an alloy of the weight of 1020 (1000 silver and 20 copper) has for its standard 980·4 in both tables. If it always contains in the same weight 4 more silver and consequently 4 less copper, its standard would be 984·3, and would be found in the 'Salt' table at the intersection of the column 4, and the horizontal line 1020. If, on the contrary, it contains 4 less of silver and 4 more of copper, its standard will be 976·5, and will be found in the 'Nitrate of Silver' table, at the intersection of the column 4, and the horizontal line 1020.

# TABLES

## FOR DETERMINING THE STANDARD OF ANY SILVER ALLOY BY EMPLOYING AN AMOUNT OF ALLOY ALWAYS APPROXIMATIVELY CONTAINING THE SAME AMOUNT OF SILVER.

*Tables for Determining the Standard of any Silver approximatively containing*

| | NITRATE OF | | | | |
|---|---|---|---|---|---|
| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
| 1000 | 1000·0 | 999·0 | 998·0 | 997·0 | 996·0 |
| 1005 | 995·0 | 994·0 | 993·0 | 992·0 | 991·0 |
| 1010 | 990·1 | 989·1 | 988·1 | 987·1 | 986·1 |
| 1015 | 985·2 | 984·2 | 983·2 | 982·3 | 981·3 |
| 1020 | 980·4 | 979·4 | 978·4 | 977·4 | 976·5 |
| 1025 | 975·6 | 974·6 | 973·7 | 972·7 | 971·7 |
| 1030 | 970·9 | 969·9 | 968·9 | 968·0 | 967·0 |
| 1035 | 966·2 | 965·2 | 964·2 | 963·3 | 962·3 |
| 1040 | 961·5 | 960·6 | 959·6 | 958·6 | 957·7 |
| 1045 | 956·9 | 956·0 | 955·0 | 954·1 | 953·1 |
| 1050 | 952·4 | 951·4 | 950·5 | 949·5 | 948·6 |
| 1055 | 947·9 | 946·9 | 946·0 | 945·0 | 944·1 |
| 1060 | 943·4 | 942·4 | 941·5 | 940·6 | 939·6 |
| 1065 | 939·0 | 938·0 | 937·1 | 936·1 | 935·2 |
| 1070 | 934·6 | 933·6 | 932·7 | 931·8 | 930·8 |
| 1075 | 930·2 | 929·3 | 928·4 | 927·4 | 926·5 |
| 1080 | 925·9 | 925·0 | 924·1 | 923·1 | 922·2 |
| 1085 | 921·7 | 920·7 | 919·8 | 918·9 | 918·0 |
| 1090 | 917·4 | 916·5 | 915·6 | 914·7 | 913·8 |
| 1095 | 913·2 | 912·3 | 911·4 | 910·5 | 909·6 |
| 1100 | 909·1 | 908·2 | 907·3 | 906·4 | 905·4 |
| 1105 | 905·0 | 904·1 | 903·2 | 902·3 | 901·4 |
| 1110 | 900·9 | 900·0 | 899·1 | 898·2 | 897·3 |
| 1115 | 896·9 | 896·0 | 895·1 | 894·2 | 893·3 |
| 1120 | 892·9 | 892·0 | 891·1 | 890·2 | 889·3 |
| 1125 | 888·9 | 888·0 | 887·1 | 886·2 | 885·3 |
| 1130 | 885·0 | 884·1 | 883·2 | 882·3 | 881·4 |
| 1135 | 881·1 | 880·2 | 879·3 | 878·4 | 877·5 |
| 1140 | 877·2 | 876·3 | 875·4 | 874·6 | 873·7 |
| 1145 | 873·4 | 872·5 | 871·6 | 870·7 | 869·9 |
| 1150 | 869·6 | 868·7 | 867·8 | 867·0 | 866·1 |
| 1155 | 865·8 | 864·9 | 864·1 | 863·2 | 862·3 |
| 1160 | 862·1 | 861·2 | 860·3 | 859·5 | 858·6 |
| 1165 | 858·4 | 857·5 | 856·6 | 855·8 | 854·9 |
| 1170 | 854·7 | 853·8 | 853·0 | 852·1 | 851·3 |
| 1175 | 851·1 | 850·2 | 849·4 | 848·5 | 847·7 |
| 1180 | 847·5 | 846·6 | 845·8 | 844·9 | 844·1 |
| 1185 | 843·9 | 843·0 | 842·2 | 841·3 | 840·5 |

*Alloy by employing an Amount of Alloy always the same Amount of Silver.*

| SILVER. | | | | | |
|---|---|---|---|---|---|
| 5. | 6. | 7. | 8. | 9. | 10. |
| 995·0 | 994·0 | 993·0 | 992·0 | 991·0 | 990·0 |
| 990·0 | 989·0 | 988·1 | 987·1 | 986·1 | 985·1 |
| 985·1 | 984·2 | 983·2 | 982·2 | 981·2 | 980·2 |
| 980·3 | 979·3 | 978·3 | 977·3 | 976·4 | 975·4 |
| 975·5 | 974·5 | 973·5 | 972·5 | 971·6 | 970·6 |
| 970·7 | 969·8 | 968·8 | 967·8 | 966·8 | 965·8 |
| 966·0 | 965·0 | 964·1 | 963·1 | 962·1 | 961·2 |
| 961·3 | 960·4 | 959·4 | 958·4 | 957·5 | 956·5 |
| 956·7 | 955·8 | 954·8 | 953·8 | 952·9 | 951·9 |
| 952·1 | 951·2 | 950·2 | 949·3 | 948·3 | 947·4 |
| 947·6 | 946·7 | 945·7 | 944·8 | 943·8 | 942·9 |
| 943·1 | 942·2 | 941·2 | 940·3 | 939·3 | 938·4 |
| 938·7 | 937·7 | 936·8 | 935·8 | 934·9 | 934·0 |
| 934·3 | 933·3 | 932·4 | 931·4 | 930·5 | 929·6 |
| 929·9 | 929·0 | 928·0 | 927·1 | 926·2 | 925·2 |
| 925·6 | 924·7 | 923·7 | 922·8 | 921·9 | 920·9 |
| 921·3 | 920·4 | 919·4 | 918·5 | 917·6 | 916·7 |
| 917·0 | 916·1 | 915·2 | 914·3 | 913·4 | 912·4 |
| 912·8 | 911·9 | 911·0 | 910·1 | 909·2 | 908·3 |
| 908·7 | 907·8 | 906·8 | 905·9 | 905·0 | 904·1 |
| 904·5 | 903·6 | 902·7 | 901·8 | 900·9 | 900·0 |
| 900·4 | 899·5 | 898·6 | 897·7 | 896·8 | 895·9 |
| 896·4 | 895·5 | 894·6 | 893·7 | 892·8 | 891·9 |
| 892·4 | 891·5 | 890·6 | 889·7 | 888·8 | 887·9 |
| 888·4 | 887·5 | 886·6 | 885·7 | 884·8 | 883·9 |
| 884·4 | 883·6 | 882·7 | 881·8 | 880·9 | 880·0 |
| 880·5 | 879·6 | 878·8 | 877·9 | 877·0 | 876·1 |
| 876·7 | 875·8 | 874·9 | 874·0 | 873·1 | 872·3 |
| 872·8 | 871·9 | 871·0 | 870·2 | 869·3 | 868·4 |
| 869·0 | 868·1 | 867·2 | 866·4 | 865·5 | 864·6 |
| 865·2 | 864·3 | 863·5 | 862·6 | 861·7 | 860·9 |
| 861·5 | 860·6 | 859·7 | 858·9 | 858·0 | 857·1 |
| 857·8 | 856·9 | 856·0 | 855·2 | 854·3 | 853·4 |
| 854·1 | 853·2 | 852·4 | 851·5 | 850·6 | 849·8 |
| 850·4 | 849·6 | 848·7 | 847·9 | 847·0 | 846·1 |
| 846·8 | 846·0 | 845·1 | 844·3 | 843·4 | 842·5 |
| 843·2 | 842·4 | 841·5 | 840·7 | 839·8 | 839·0 |
| 839·7 | 838·8 | 838·0 | 837·1 | 836·3 | 835·4 |

NITRATE OF

| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
|---|---|---|---|---|---|
| 1190 | 840·3 | 849·5 | 838·7 | 837·8 | 837·0 |
| 1195 | 836·8 | 836·0 | 835·1 | 834·3 | 833·5 |
| 1200 | 833·3 | 832·5 | 831·7 | 830·8 | 830·0 |
| 1205 | 829·9 | 829·0 | 828·2 | 827·4 | 826·6 |
| 1210 | 826·4 | 825·6 | 824·8 | 824·0 | 823·1 |
| 1215 | 823·0 | 822·2 | 821·4 | 820·6 | 819·7 |
| 1220 | 819·7 | 818·8 | 818·0 | 817·2 | 816·4 |
| 1225 | 816·3 | 815·5 | 814·7 | 813·9 | 813·1 |
| 1230 | 813·0 | 812·2 | 811·4 | 810·6 | 809·8 |
| 1235 | 809·7 | 808·9 | 808·1 | 807·3 | 806·5 |
| 1240 | 806·5 | 805·6 | 804·8 | 804·0 | 803·2 |
| 1245 | 803·2 | 802·4 | 801·6 | 800·8 | 800·0 |
| 1250 | 800·0 | 799·2 | 798·4 | 797·6 | 796·8 |
| 1255 | 796·8 | 796·0 | 795·2 | 794·4 | 793·6 |
| 1260 | 793·6 | 792·9 | 792·1 | 791·3 | 790·5 |
| 1265 | 790·5 | 789·7 | 788·9 | 788·1 | 787·3 |
| 1270 | 787·4 | 786·6 | 785·8 | 785·0 | 784·2 |
| 1275 | 784·3 | 783·5 | 782·7 | 782·0 | 781·2 |
| 1280 | 781·2 | 780·5 | 779·7 | 778·9 | 778·1 |
| 1285 | 778·2 | 777·4 | 776·6 | 775·9 | 775·1 |
| 1290 | 775·2 | 774·4 | 773·6 | 772·9 | 772·1 |
| 1295 | 772·2 | 771·4 | 770·7 | 769·9 | 769·1 |
| 1300 | 769·2 | 768·5 | 767·7 | 766·9 | 766·1 |
| 1305 | 766·3 | 765·5 | 764·7 | 764·0 | 763·2 |
| 1310 | 763·4 | 762·6 | 761·8 | 761·1 | 760·3 |
| 1315 | 760·5 | 759·7 | 758·9 | 758·2 | 757·4 |
| 1320 | 757·6 | 756·8 | 756·1 | 755·3 | 754·5 |
| 1325 | 754·7 | 754·0 | 753·2 | 752·4 | 751·7 |
| 1330 | 751·9 | 751·1 | 750·4 | 749·6 | 748·9 |
| 1335 | 749·1 | 748·3 | 747·6 | 746·8 | 746·1 |
| 1340 | 746·3 | 745·5 | 744·8 | 744·0 | 743·3 |
| 1345 | 743·5 | 742·7 | 742·0 | 741·3 | 740·5 |
| 1350 | 740·7 | 740·0 | 739·3 | 738·5 | 737·8 |
| 1355 | 738·0 | 737·3 | 736·5 | 735·8 | 735·1 |
| 1360 | 735·3 | 734·6 | 733·8 | 733·1 | 732·4 |
| 1365 | 732·6 | 731·9 | 731·1 | 730·4 | 729·7 |
| 1370 | 729·9 | 729·2 | 728·5 | 727·7 | 727·0 |
| 1375 | 727·3 | 726·5 | 725·8 | 725·1 | 724·4 |
| 1380 | 724·6 | 723·9 | 723·2 | 722·5 | 721·7 |
| 1385 | 722·0 | 721·3 | 720·6 | 719·9 | 719·1 |
| 1390 | 719·4 | 718·7 | 718·0 | 717·3 | 716·5 |
| 1395 | 716·8 | 716·1 | 715·4 | 714·7 | 714·0 |
| 1400 | 714·3 | 713·6 | 712·9 | 712·1 | 711·4 |

**SILVER**—*continued.*

| 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|
| 836·1 | 835·3 | 834·5 | 833·6 | 832·8 | 831·9 |
| 832·6 | 831·8 | 831·0 | 830·1 | 829·3 | 828·4 |
| 829·2 | 828·3 | 827·5 | 826·7 | 825·8 | 825·0 |
| 825·7 | 824·9 | 824·1 | 823·2 | 822·4 | 821·6 |
| 822·3 | 821·5 | 820·7 | 819·8 | 819·0 | 818·2 |
| 818·9 | 818·1 | 817·3 | 816·5 | 815·6 | 814·8 |
| 815·6 | 814·7 | 813·9 | 813·1 | 812·3 | 811·5 |
| 812·2 | 811·4 | 810·6 | 809·8 | 809·0 | 808·2 |
| 808·9 | 808·1 | 807·3 | 806·5 | 805·7 | 804·9 |
| 805·7 | 804·9 | 804·0 | 803·2 | 802·4 | 801·6 |
| 802·4 | 801·6 | 800·8 | 800·0 | 799·2 | 798·4 |
| 799·2 | 798·4 | 797·6 | 796·8 | 796·0 | 795·2 |
| 796·0 | 795·2 | 794·4 | 793·6 | 792·8 | 792·0 |
| 792·8 | 792·0 | 791·2 | 790·4 | 789·6 | 788·8 |
| 789·7 | 788·9 | 788·1 | 787·3 | 786·5 | 785·7 |
| 786·6 | 785·8 | 785·0 | 784·2 | 783·4 | 782·6 |
| 783·5 | 782·7 | 781·9 | 781·1 | 780·3 | 779·5 |
| 780·4 | 779·6 | 778·8 | 778·0 | 777·3 | 776·5 |
| 777·3 | 776·6 | 775·8 | 775·0 | 774·2 | 773·4 |
| 774·3 | 773·5 | 772·8 | 772·0 | 771·2 | 770·4 |
| 771·3 | 770·5 | 769·8 | 769·0 | 768·2 | 767·4 |
| 768·3 | 767·6 | 766·8 | 766·0 | 765·2 | 764·5 |
| 765·4 | 764·6 | 763·8 | 763·1 | 762·3 | 761·5 |
| 762·4 | 761·7 | 760·9 | 760·1 | 759·4 | 758·6 |
| 759·5 | 758·8 | 758·0 | 757·2 | 756·5 | 755·7 |
| 756·6 | 755·9 | 755·1 | 754·4 | 753·6 | 752·8 |
| 753·8 | 753·0 | 752·3 | 751·5 | 750·8 | 750·0 |
| 750·9 | 750·2 | 749·4 | 748·7 | 747·9 | 747·2 |
| 748·1 | 747·4 | 746·6 | 745·9 | 745·1 | 744·4 |
| 745·3 | 744·6 | 743·8 | 743·1 | 742·3 | 741·6 |
| 742·5 | 741·8 | 741·0 | 740·3 | 739·5 | 738·8 |
| 789·8 | 739·0 | 738·3 | 737·5 | 736·8 | 736·1 |
| 737·0 | 736·3 | 735·6 | 734·8 | 734·1 | 733·3 |
| 734·3 | 733·6 | 732·8 | 732·1 | 731·4 | 730·6 |
| 731·6 | 730·9 | 730·1 | 729·4 | 728·7 | 727·9 |
| 728·9 | 728·2 | 727·5 | 726·7 | 726·0 | 725·3 |
| 726·3 | 725·5 | 724·8 | 724·1 | 723·4 | 722·6 |
| 723·6 | 722·9 | 722·2 | 721·4 | 720·7 | 720·0 |
| 721·0 | 720·3 | 719·6 | 718·8 | 718·1 | 717·4 |
| 718·4 | 717·7 | 717·0 | 716·2 | 715·5 | 714·8 |
| 715·8 | 715·1 | 714·4 | 713·7 | 712·9 | 712·2 |
| 713·3 | 712·5 | 711·8 | 711·1 | 710·4 | 709·7 |
| 710·7 | 710·0 | 709·3 | 708·6 | 707·9 | 707·1 |

NITRATE OF

| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
|---|---|---|---|---|---|
| 1405 | 711·7 | 711·0 | 710·3 | 709·6 | 708·9 |
| 1410 | 709·2 | 708·5 | 707·8 | 707·1 | 706·4 |
| 1415 | 706·7 | 706·0 | 705·3 | 704·6 | 703·9 |
| 1420 | 704·2 | 703·5 | 702·8 | 702·1 | 701·4 |
| 1425 | 701·8 | 701·0 | 700·3 | 699·6 | 698·9 |
| 1430 | 699·3 | 698·6 | 697·9 | 697·2 | 696·5 |
| 1435 | 696·9 | 696·2 | 695·5 | 694·8 | 694·1 |
| 1440 | 694·4 | 693·7 | 693·1 | 692·4 | 691·7 |
| 1445 | 692·0 | 691·3 | 690·7 | 690·0 | 689·3 |
| 1450 | 689·7 | 689·0 | 688·3 | 687·6 | 686·9 |
| 1455 | 687·3 | 686·6 | 685·9 | 685·2 | 684·5 |
| 1460 | 684·9 | 684·2 | 683·6 | 682·9 | 682·2 |
| 1465 | 682·6 | 681·9 | 681·2 | 680·6 | 679·9 |
| 1470 | 680·3 | 679·6 | 678·9 | 678·2 | 677·5 |
| 1475 | 678·0 | 677·3 | 676·6 | 675·9 | 675·2 |
| 1480 | 675·7 | 675·0 | 674·3 | 673·6 | 673·0 |
| 1485 | 673·4 | 672·7 | 672·0 | 671·4 | 670·7 |
| 1490 | 671·1 | 670·5 | 669·8 | 669·1 | 668·5 |
| 1495 | 668·9 | 668·2 | 667·6 | 666·9 | 666·2 |
| 1500 | 666·7 | 666·0 | 665·3 | 664·7 | 664·0 |
| 1505 | 664·5 | 663·8 | 663·1 | 662·5 | 661·8 |
| 1510 | 662·3 | 661·6 | 660·9 | 660·3 | 659·6 |
| 1515 | 660·1 | 659·4 | 658·7 | 658·1 | 657·4 |
| 1520 | 657·9 | 657·2 | 656·6 | 655·9 | 655·3 |
| 1525 | 655·7 | 655·1 | 654·4 | 653·8 | 653·1 |
| 1530 | 653·6 | 652·9 | 652·3 | 651·6 | 651·0 |
| 1535 | 651·5 | 650·8 | 650·2 | 649·5 | 648·9 |
| 1540 | 649·4 | 648·7 | 648·0 | 647·4 | 646·7 |
| 1545 | 647·2 | 646·6 | 645·9 | 645·3 | 644·7 |
| 1550 | 645·2 | 644·5 | 643·9 | 643·2 | 642·6 |
| 1555 | 643·1 | 642·4 | 641·8 | 641·2 | 640·5 |
| 1560 | 641·0 | 640·4 | 639·7 | 639·1 | 638·5 |
| 1565 | 639·0 | 638·3 | 637·7 | 637·1 | 636·4 |
| 1570 | 636·9 | 636·3 | 635·7 | 635·0 | 634·4 |
| 1575 | 634·9 | 634·3 | 633·6 | 633·0 | 632·4 |
| 1580 | 632·9 | 632·3 | 631·6 | 631·0 | 630·4 |
| 1585 | 630·9 | 630·3 | 629·6 | 629·0 | 628·4 |
| 1590 | 628·9 | 623·3 | 627·7 | 627·0 | 626·4 |
| 1595 | 627·0 | 626·3 | 625·7 | 625·1 | 624·4 |
| 1600 | 625·0 | 624·4 | 623·7 | 623·1 | 622·5 |
| 1605 | 623·1 | 622·4 | 621·8 | 621·2 | 620·6 |
| 1610 | 621·1 | 620·5 | 619·9 | 619·2 | 618·6 |
| 1615 | 619·2 | 618·6 | 618·0 | 617·3 | 616·7 |

SILVER—*continued.*

| 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|
| 708·2 | 707·5 | 706·8 | 706·0 | 705·3 | 704·6 |
| 705·7 | 705·0 | 704·3 | 703·5 | 702·8 | 702·1 |
| 703·2 | 702·5 | 701·8 | 701·1 | 700·3 | 699·6 |
| 700·7 | 700·0 | 699·3 | 698·6 | 697·9 | 697·2 |
| 698·2 | 697·5 | 696·8 | 696·1 | 695·4 | 694·7 |
| 695·8 | 695·1 | 694·4 | 693·7 | 693·0 | 692·3 |
| 693·4 | 692·7 | 692·0 | 691·3 | 690·6 | 689·9 |
| 691·0 | 690·3 | 689·6 | 688·9 | 688·2 | 687·5 |
| 688·6 | 687·9 | 687·2 | 686·5 | 685·8 | 685·1 |
| 686·2 | 685·5 | 684·8 | 684·1 | 683·4 | 682·8 |
| 683·8 | 683·2 | 682·5 | 681·8 | 681·1 | 680·4 |
| 681·5 | 680·8 | 680·1 | 679·4 | 678·8 | 678·1 |
| 679·2 | 678·5 | 677·8 | 677·1 | 676·4 | 675·8 |
| 676·9 | 676·2 | 675·5 | 674·8 | 674·1 | 673·5 |
| 674·6 | 673·9 | 673·2 | 672·5 | 671·9 | 671·2 |
| 672·3 | 671·6 | 670·9 | 670·3 | 669·6 | 668·9 |
| 670·0 | 669·4 | 668·7 | 668·0 | 667·3 | 666·7 |
| 667·8 | 667·1 | 666·4 | 665·8 | 665·1 | 664·4 |
| 665·5 | 664·9 | 664·2 | 663·5 | 662·9 | 662·2 |
| 663·3 | 662·7 | 662·0 | 661·3 | 660·7 | 660·0 |
| 661·1 | 660·5 | 659·8 | 659·1 | 658·5 | 657·8 |
| 658·9 | 658·3 | 657·6 | 656·9 | 656·3 | 655·6 |
| 656·8 | 656·1 | 655·4 | 654·8 | 654·1 | 653·5 |
| 654·6 | 653·9 | 653·3 | 652·6 | 652·0 | 651·3 |
| 652·5 | 651·8 | 651·1 | 650·5 | 649·8 | 649·2 |
| 650·3 | 649·7 | 649·0 | 648·4 | 647·7 | 647·1 |
| 648·2 | 647·6 | 646·9 | 646·2 | 645·6 | 644·9 |
| 646·1 | 645·4 | 644·8 | 644·2 | 643·5 | 642·9 |
| 644·0 | 643·4 | 642·7 | 642·1 | 641·4 | 640·8 |
| 641·9 | 641·3 | 640·6 | 640·0 | 639·3 | 638·7 |
| 639·9 | 639·2 | 638·6 | 637·9 | 637·3 | 636·7 |
| 637·8 | 637·2 | 636·5 | 635·9 | 635·3 | 634·6 |
| 635·8 | 635·1 | 634·5 | 633·9 | 633·2 | 632·6 |
| 633·8 | 633·1 | 632·5 | 631·8 | 631·2 | 630·6 |
| 631·7 | 631·1 | 630·5 | 629·8 | 629·2 | 628·6 |
| 629·7 | 629·1 | 628·5 | 627·8 | 627·2 | 626·6 |
| 627·8 | 627·1 | 626·5 | 625·9 | 625·2 | 624·6 |
| 625·8 | 625·2 | 624·5 | 623·9 | 623·3 | 622·6 |
| 623·8 | 623·2 | 622·6 | 621·9 | 621·3 | 620·7 |
| 621·9 | 621·2 | 620·6 | 620·0 | 619·4 | 618·7 |
| 619·9 | 619·3 | 618·7 | 618·1 | 617·4 | 616·1 |
| 618·0 | 617·4 | 616·8 | 616·1 | 615·5 | 614·9 |
| 616·1 | 615·5 | 614·9 | 614·2 | 613·6 | 613·0 |

| | NITRATE OF | | | | |
|---|---|---|---|---|---|
| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
| 1620 | 617·3 | 616·7 | 616·0 | 615·4 | 614·8 |
| 1625 | 615·4 | 614·8 | 614·1 | 613·5 | 612·9 |
| 1630 | 613·5 | 612·9 | 612·3 | 611·7 | 611·0 |
| 1635 | 611·6 | 611·0 | 610·4 | 609·8 | 609·2 |
| 1640 | 609·8 | 609·1 | 608·5 | 607·9 | 607·3 |
| 1645 | 607·9 | 607·3 | 606·7 | 606·1 | 605·5 |
| 1650 | 606·1 | 605·4 | 604·8 | 604·2 | 603·6 |
| 1655 | 604·2 | 603·6 | 603·0 | 602·4 | 601·8 |
| 1660 | 602·4 | 601·8 | 601·2 | 600·6 | 600·0 |
| 1665 | 600·6 | 600·0 | 599·4 | 598·8 | 598·2 |
| 1670 | 598·8 | 598·2 | 597·6 | 597·0 | 596·4 |
| 1675 | 597·0 | 596·4 | 595·8 | 595·2 | 594·6 |
| 1680 | 595·2 | 594·6 | 594·0 | 593·4 | 592·9 |
| 1685 | 593·5 | 592·9 | 592·3 | 591·7 | 591·1 |
| 1690 | 591·7 | 591·1 | 590·5 | 589·9 | 589·3 |
| 1695 | 590·0 | 589·4 | 588·8 | 588·2 | 587·6 |
| 1700 | 588·2 | 587·6 | 587·1 | 586·5 | 585·9 |
| 1705 | 586·5 | 585·9 | 585·3 | 584·7 | 584·2 |
| 1710 | 584·8 | 584·2 | 583·6 | 583·0 | 582·5 |
| 1715 | 583·1 | 582·5 | 581·9 | 581·3 | 580·8 |
| 1720 | 581·4 | 580·8 | 580·2 | 579·6 | 579·1 |
| 1725 | 579·7 | 579·1 | 578·5 | 578·0 | 577·4 |
| 1730 | 578·0 | 577·5 | 576·9 | 576·3 | 575·7 |
| 1735 | 576·4 | 575·8 | 575·2 | 574·6 | 574·1 |
| 1740 | 574·7 | 574·1 | 573·6 | 573·0 | 572·4 |
| 1745 | 573·1 | 572·5 | 571·9 | 571·3 | 570·8 |
| 1750 | 571·4 | 570·9 | 570·3 | 569·7 | 569·1 |
| 1755 | 569·8 | 569·2 | 568·7 | 568·1 | 567·5 |
| 1760 | 568·2 | 567·6 | 567·0 | 566·5 | 565·9 |
| 1765 | 566·6 | 566·0 | 565·4 | 564·9 | 564·3 |
| 1770 | 565·0 | 564·4 | 563·8 | 563·3 | 562·7 |
| 1775 | 563·4 | 562·8 | 562·2 | 561·7 | 561·1 |
| 1780 | 561·8 | 561·2 | 560·7 | 560·1 | 559·5 |
| 1785 | 560·2 | 559·7 | 559·1 | 558·5 | 558·0 |
| 1790 | 558·7 | 558·1 | 557·5 | 557·0 | 556·4 |
| 1795 | 557·1 | 556·5 | 556·0 | 555·4 | 554·9 |
| 1800 | 555·6 | 555·0 | 554·4 | 553·9 | 553·3 |
| 1805 | 554·0 | 553·5 | 552·9 | 552·3 | 551·8 |
| 1810 | 552·5 | 551·9 | 551·4 | 550·8 | 550·3 |
| 1815 | 551·0 | 550·4 | 549·9 | 549·3 | 548·8 |
| 1820 | 549·4 | 548·9 | 548·3 | 547·8 | 547·2 |
| 1825 | 547·9 | 547·4 | 546·8 | 546·3 | 545·7 |
| 1830 | 546·4 | 545·9 | 545·4 | 544·8 | 544·3 |

SILVER—*continued.*

| 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|
| 614·2 | 613·6 | 613·0 | 612·3 | 611·7 | 611·1 |
| 612·3 | 611·7 | 611·1 | 610·5 | 609·8 | 609·2 |
| 610·4 | 609·8 | 609·2 | 608·6 | 608·0 | 607·4 |
| 608·6 | 607·9 | 607·3 | 606·7 | 606·1 | 605·5 |
| 606·7 | 606·1 | 605·5 | 604·9 | 604·3 | 603·7 |
| 604·9 | 604·3 | 603·6 | 603·0 | 602·4 | 601·8 |
| 603·0 | 602·4 | 601·8 | 601·2 | 600·6 | 600·0 |
| 601·2 | 600·6 | 600·0 | 599·4 | 598·8 | 598·2 |
| 599·4 | 598·8 | 598·2 | 597·6 | 597·0 | 596·4 |
| 597·6 | 597·0 | 596·4 | 595·8 | 595·2 | 594·6 |
| 595·8 | 595·2 | 594·6 | 594·0 | 593·4 | 592·8 |
| 594·0 | 593·4 | 592·8 | 592·2 | 591·6 | 591·0 |
| 592·3 | 591·7 | 591·1 | 590·5 | 589·9 | 589·3 |
| 590·5 | 589·9 | 589·3 | 588·7 | 588·1 | 587·5 |
| 588·8 | 588·2 | 587·6 | 587·0 | 586·4 | 585·8 |
| 587·0 | 586·4 | 585·8 | 585·2 | 584·7 | 584·1 |
| 585·3 | 584·7 | 584·1 | 583·5 | 582·9 | 582·3 |
| 583·6 | 583·0 | 582·4 | 581·8 | 581·2 | 580·6 |
| 581·9 | 581·3 | 580·7 | 580·1 | 579·5 | 578·9 |
| 580·2 | 579·6 | 579·0 | 578·4 | 577·8 | 577·3 |
| 578·5 | 577·9 | 577·3 | 576·7 | 576·2 | 575·6 |
| 576·8 | 576·2 | 575·6 | 575·1 | 574·5 | 573·9 |
| 575·1 | 574·6 | 574·0 | 573·4 | 572·8 | 572·2 |
| 573·5 | 572·9 | 572·3 | 571·8 | 571·2 | 570·6 |
| 571·8 | 571·3 | 570·7 | 570·1 | 569·5 | 569·0 |
| 570·2 | 569·6 | 569·0 | 568·5 | 567·9 | 567·3 |
| 568·6 | 568·0 | 567·4 | 566·9 | 566·3 | 565·7 |
| 566·9 | 566·4 | 565·8 | 565·2 | 564·7 | 564·1 |
| 565·3 | 564·8 | 564·2 | 563·6 | 563·1 | 562·5 |
| 563·7 | 563·2 | 562·6 | 562·0 | 561·5 | 560·9 |
| 562·1 | 561·6 | 561·0 | 560·4 | 559·9 | 559·3 |
| 560·6 | 560·0 | 559·4 | 558·9 | 558·3 | 557·7 |
| 559·0 | 558·4 | 557·9 | 557·3 | 556·7 | 556·2 |
| 557·4 | 556·9 | 556·3 | 555·7 | 555·2 | 554·6 |
| 555·9 | 555·3 | 554·7 | 554·2 | 553·6 | 553·1 |
| 554·3 | 553·8 | 553·2 | 552·6 | 552·1 | 551·5 |
| 552·8 | 552·2 | 551·7 | 551·1 | 550·6 | 550·0 |
| 551·2 | 550·7 | 550·1 | 549·5 | 549·0 | 548·5 |
| 549·7 | 549·2 | 548·6 | 548·1 | 547·5 | 547·0 |
| 548·2 | 547·7 | 547·1 | 546·6 | 546·0 | 545·5 |
| 546·7 | 546·2 | 545·6 | 545·1 | 544·5 | 544·0 |
| 545·2 | 544·7 | 544·1 | 543·6 | 543·0 | 542·5 |
| 543·7 | 543·2 | 542·6 | 542·1 | 541·5 | 541·0 |

NITRATE OF

| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
|---|---|---|---|---|---|
| 1835 | 545·0 | 544·4 | 543·9 | 543·3 | 542·8 |
| 1840 | 543·5 | 542·9 | 542·4 | 541·8 | 541·3 |
| 1845 | 542·0 | 541·5 | 540·9 | 540·4 | 539·8 |
| 1850 | 540·5 | 540·0 | 539·5 | 538·9 | 538·4 |
| 1855 | 539·1 | 538·5 | 538·0 | 537·5 | 536·9 |
| 1860 | 537·6 | 537·1 | 536·6 | 536·0 | 535·5 |
| 1865 | 536·2 | 535·7 | 535·1 | 534·6 | 534·0 |
| 1870 | 534·8 | 534·2 | 533·7 | 533·2 | 532·6 |
| 1875 | 533·3 | 532·8 | 532·3 | 531·7 | 531·2 |
| 1880 | 531·9 | 531·4 | 530·8 | 530·3 | 529·8 |
| 1885 | 530·5 | 530·0 | 529·4 | 528·9 | 528·4 |
| 1890 | 529·1 | 528·6 | 528·0 | 527·5 | 527·0 |
| 1895 | 527·7 | 527·2 | 526·6 | 526·1 | 525·6 |
| 1900 | 526·3 | 525·8 | 525·3 | 524·7 | 524·2 |
| 1905 | 524·9 | 524·4 | 523·9 | 523·4 | 522·8 |
| 1910 | 523·6 | 523·0 | 522·5 | 522·0 | 521·5 |
| 1915 | 522·2 | 521·7 | 521·1 | 520·6 | 520·1 |
| 1920 | 520·8 | 520·3 | 519·8 | 519·3 | 518·7 |
| 1925 | 519·5 | 519·0 | 518·4 | 517·9 | 517·4 |
| 1930 | 518·1 | 517·6 | 517·1 | 516·6 | 516·1 |
| 1935 | 516·8 | 516·3 | 515·8 | 515·2 | 514·7 |
| 1940 | 515·5 | 514·9 | 514·4 | 513·9 | 513·4 |
| 1945 | 514·1 | 513·6 | 513·1 | 512·6 | 512·1 |
| 1950 | 512·8 | 512·3 | 511·8 | 511·3 | 510·8 |
| 1955 | 511·5 | 511·0 | 510·5 | 510·0 | 509·5 |
| 1960 | 510·2 | 509·7 | 509·2 | 508·7 | 508·2 |
| 1965 | 508·9 | 508·4 | 507·9 | 507·4 | 506·9 |
| 1970 | 507·6 | 507·1 | 506·6 | 506·1 | 505·6 |
| 1975 | 506·3 | 505·8 | 505·3 | 504·8 | 504·3 |
| 1980 | 505·0 | 504·5 | 504·0 | 503·5 | 503·0 |
| 1985 | 503·8 | 503·3 | 502·8 | 502·3 | 501·8 |
| 1990 | 502·5 | 502·0 | 501·5 | 501·0 | 500·5 |
| 1995 | 501·3 | 500·7 | 500·2 | 499·7 | 499·2 |
| 2000 | 500·0 | 499·5 | 499·0 | 498·5 | 498·0 |

SILVER—*continued.*

| 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|
| 542·2 | 541·7 | 541·1 | 540·6 | 540·0 | 539·5 |
| 540·8 | 540·2 | 539·7 | 539·1 | 538·6 | 538·0 |
| 539·3 | 538·7 | 538·2 | 537·7 | 537·1 | 536·6 |
| 537·8 | 537·3 | 536·8 | 536·2 | 535·7 | 535·1 |
| 536·4 | 535·8 | 535·3 | 534·8 | 534·2 | 533·7 |
| 534·9 | 534·4 | 533·9 | 533·3 | 532·8 | 532·3 |
| 533·5 | 533·0 | 532·4 | 531·9 | 531·4 | 530·8 |
| 532·1 | 531·5 | 531·0 | 530·5 | 529·9 | 529·4 |
| 530·7 | 530·1 | 529·6 | 529·1 | 528·5 | 528·0 |
| 529·3 | 528·7 | 528·2 | 527·7 | 527·1 | 526·6 |
| 527·8 | 527·3 | 526·8 | 526·3 | 525·7 | 525·2 |
| 526·5 | 525·9 | 525·4 | 524·9 | 524·3 | 523·8 |
| 525·1 | 524·5 | 524·0 | 523·5 | 523·0 | 522·4 |
| 523·7 | 523·2 | 522·6 | 522·1 | 521·6 | 521·0 |
| 522·3 | 521·8 | 521·3 | 520·7 | 520·2 | 519·7 |
| 520·9 | 520·4 | 519·9 | 519·4 | 518·8 | 518·3 |
| 519·6 | 519·1 | 518·5 | 518·0 | 517·5 | 517·0 |
| 518·2 | 517·7 | 517·2 | 516·7 | 516·1 | 515·6 |
| 516·9 | 516·4 | 515·8 | 515·3 | 514·8 | 514·3 |
| 515·5 | 515·0 | 514·5 | 514·0 | 513·5 | 512·9 |
| 514·2 | 513·7 | 513·2 | 512·7 | 512·1 | 511·6 |
| 512·9 | 512·4 | 511·9 | 511·3 | 510·8 | 510·3 |
| 511·6 | 511·0 | 510·5 | 510·0 | 509·5 | 509·0 |
| 510·3 | 509·7 | 509·2 | 508·7 | 508·2 | 507·7 |
| 508·9 | 508·4 | 507·9 | 507·4 | 506·9 | 506·4 |
| 507·6 | 507·1 | 506·6 | 506·1 | 505·6 | 505·1 |
| 506·4 | 505·8 | 505·3 | 504·8 | 504·3 | 503·8 |
| 505·1 | 504·6 | 504·1 | 503·5 | 503·0 | 502·5 |
| 503·8 | 503·3 | 502·8 | 502·3 | 501·8 | 501·3 |
| 502·5 | 509·0 | 501·5 | 501·0 | 500·5 | 500·0 |
| 501·3 | 500·8 | 500·2 | 499·7 | 499·2 | 498·7 |
| 500·0 | 499·5 | 499·0 | 498·5 | 498·0 | 497·5 |
| 498·7 | 498·2 | 497·7 | 497·2 | 496·7 | 496·2 |
| 497·5 | 497·0 | 496·5 | 496·0 | 495·5 | 495·0 |

*Tables for Determining the Standard of any Silver approximatively containing*

| | | | | | COMMON |
|---|---|---|---|---|---|
| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
| 1000 | 1000·0 | | | | |
| 1005 | 995·0 | 996·0 | 997·0 | 998·0 | 999·0 |
| 1010 | 990·1 | 991·1 | 992·1 | 993·1 | 994·1 |
| 1015 | 985·2 | 986·2 | 987·2 | 988·2 | 989·2 |
| 1020 | 980·4 | 981·4 | 982·4 | 983·3 | 984·3 |
| 1025 | 975·6 | 976·6 | 977·6 | 978·5 | 979·5 |
| 1030 | 970·9 | 971·8 | 972·8 | 973·8 | 974·8 |
| 1035 | 966·2 | 967·1 | 968·1 | 969·1 | 970·0 |
| 1040 | 961·5 | 962·5 | 963·5 | 964·4 | 965·4 |
| 1045 | 956·9 | 957·9 | 958·8 | 959·8 | 960·8 |
| 1050 | 952·4 | 953·3 | 954·3 | 955·2 | 956·2 |
| 1055 | 947·9 | 948·8 | 949·8 | 950·7 | 951·7 |
| 1060 | 943·4 | 944·3 | 945·3 | 946·2 | 947·2 |
| 1065 | 939·0 | 939·9 | 940·8 | 941·8 | 942·7 |
| 1070 | 934·6 | 935·5 | 936·4 | 937·4 | 938·3 |
| 1075 | 930·2 | 931·2 | 932·1 | 933·0 | 933·9 |
| 1080 | 925·9 | 926·8 | 927·8 | 928·7 | 929·6 |
| 1085 | 921·7 | 922·6 | 923·5 | 924·4 | 925·3 |
| 1090 | 917·4 | 918·3 | 919·3 | 920·2 | 921·1 |
| 1095 | 913·2 | 914·2 | 915·1 | 916·0 | 917·0 |
| 1100 | 909·1 | 910·0 | 910·9 | 911·8 | 912·7 |
| 1105 | 905·0 | 905·9 | 906·8 | 907·7 | 908·6 |
| 1110 | 900·9 | 901·8 | 902·7 | 903·6 | 904·5 |
| 1115 | 896·9 | 897·8 | 898·6 | 899·5 | 900·4 |
| 1120 | 892·9 | 893·7 | 894·6 | 895·5 | 896·4 |
| 1125 | 888·9 | 889·8 | 890·7 | 891·6 | 892·4 |
| 1130 | 885·0 | 885·8 | 886·7 | 887·6 | 888·5 |
| 1135 | 881·1 | 881·9 | 882·8 | 883·7 | 884·6 |
| 1140 | 877·2 | 878·1 | 878·9 | 879·8 | 880·7 |
| 1145 | 873·4 | 874·2 | 875·1 | 876·0 | 876·9 |
| 1150 | 869·6 | 870·4 | 871·3 | 872·2 | 873·0 |
| 1155 | 865·8 | 866·7 | 867·5 | 868·4 | 869·3 |
| 1160 | 862·1 | 862·9 | 863·8 | 864·7 | 865·5 |
| 1165 | 858·4 | 859·2 | 860·1 | 860·9 | 861·8 |
| 1170 | 854·7 | 855·6 | 856·4 | 857·3 | 858·1 |
| 1175 | 851·1 | 851·9 | 852·8 | 853·6 | 854·5 |
| 1180 | 847·5 | 848·3 | 849·2 | 850·0 | 850·8 |
| 1185 | 843·9 | 844·7 | 845·6 | 846·4 | 847·3 |

*Alloy by employing an Amount of Alloy always the same Amount of Silver.*

| SALT. | | | | | |
|---|---|---|---|---|---|
| 5. | 6. | 7. | 8. | 9. | 10. |
| 1000·0 | | | | | |
| 995·0 | 996·0 | 997·0 | 998·0 | 999·0 | 1000·0 |
| 990·1 | 991·1 | 992·1 | 993·1 | 994·1 | 995·1 |
| 985·3 | 986·3 | 987·2 | 988·2 | 989·2 | 990·2 |
| 980·5 | 981·5 | 982·4 | 983·4 | 984·4 | 985·4 |
| 975·7 | 976·7 | 977·7 | 978·6 | 979·6 | 980·6 |
| 971·0 | 972·0 | 972·9 | 973·9 | 974·9 | 975·8 |
| 966·3 | 967·3 | 968·3 | 969·2 | 970·2 | 971·1 |
| 961·7 | 962·7 | 963·6 | 964·6 | 965·5 | 966·5 |
| 957·1 | 958·1 | 959·0 | 960·0 | 960·9 | 961·9 |
| 952·6 | 953·5 | 954·5 | 955·4 | 956·4 | 957·3 |
| 948·1 | 949·1 | 950·0 | 950·9 | 951·9 | 952·8 |
| 943·7 | 944·6 | 945·5 | 946·5 | 947·4 | 948·4 |
| 939·3 | 940·2 | 941·1 | 942·1 | 943·0 | 943·9 |
| 934·9 | 935·8 | 936·7 | 937·7 | 938·6 | 939·5 |
| 930·6 | 931·5 | 932·4 | 933·3 | 934·3 | 935·2 |
| 926·3 | 927·2 | 928·1 | 929·0 | 930·0 | 930·9 |
| 922·0 | 922·9 | 923·8 | 924·8 | 925·7 | 926·6 |
| 917·8 | 918·7 | 919·6 | 920·5 | 921·5 | 922·4 |
| 913·6 | 914·5 | 915·4 | 916·4 | 917·3 | 918·2 |
| 909·5 | 910·4 | 911·3 | 912·2 | 913·1 | 914·0 |
| 905·4 | 906·3 | 907·2 | 908·1 | 909·0 | 909·9 |
| 901·3 | 902·2 | 903·1 | 904·0 | 904·9 | 905·8 |
| 897·3 | 898·2 | 899·1 | 900·0 | 900·9 | 901·8 |
| 893·3 | 894·2 | 895·1 | 896·0 | 896·9 | 897·8 |
| 889·4 | 890·3 | 891·1 | 892·0 | 892·9 | 893·8 |
| 885·5 | 886·3 | 887·2 | 888·1 | 889·0 | 889·9 |
| 881·6 | 882·5 | 883·3 | 884·2 | 885·1 | 886·0 |
| 877·7 | 878·6 | 879·5 | 880·3 | 881·2 | 882·1 |
| 873·9 | 874·8 | 875·7 | 876·5 | 877·4 | 878·3 |
| 870·1 | 871·0 | 871·9 | 872·7 | 873·6 | 874·5 |
| 866·4 | 867·2 | 868·1 | 869·0 | 869·8 | 870·7 |
| 862·7 | 863·5 | 864·4 | 865·2 | 866·1 | 866·9 |
| 859·0 | 859·8 | 860·7 | 861·5 | 862·4 | 863·2 |
| 855·3 | 856·2 | 857·0 | 857·9 | 858·7 | 859·6 |
| 851·7 | 852·5 | 853·4 | 854·2 | 855·1 | 855·9 |
| 848·1 | 848·9 | 849·8 | 850·6 | 851·5 | 852·3 |

COMMON

| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
|---|---|---|---|---|---|
| 1190 | 840·3 | 841·2 | 842·0 | 842·9 | 843·7 |
| 1195 | 836·8 | 837·7 | 838·5 | 839·3 | 840·2 |
| 1200 | 833·3 | 834·2 | 835·0 | 835·8 | 836·7 |
| 1205 | 829·9 | 830·7 | 831·5 | 832·4 | 833·2 |
| 1210 | 826·4 | 827·3 | 828·1 | 828·9 | 829·7 |
| 1215 | 823·0 | 823·9 | 824·7 | 825·5 | 826·3 |
| 1220 | 819·7 | 820·5 | 821·3 | 822·1 | 822·9 |
| 1225 | 816·3 | 817·1 | 818·0 | 818·8 | 819·6 |
| 1230 | 813·0 | 813·8 | 814·6 | 815·4 | 816·3 |
| 1235 | 809·7 | 810·5 | 811·3 | 812·1 | 813·0 |
| 1240 | 806·5 | 807·3 | 808·1 | 808·9 | 809·7 |
| 1245 | 803·2 | 804·0 | 804·8 | 805·6 | 806·4 |
| 1250 | 800·0 | 800·8 | 801·6 | 802·4 | 803·2 |
| 1255 | 796·8 | 797·6 | 798·4 | 799·2 | 800·0 |
| 1260 | 793·6 | 794·4 | 795·2 | 796·0 | 796·8 |
| 1265 | 790·5 | 791·3 | 792·1 | 792·9 | 793·7 |
| 1270 | 787·4 | 788·2 | 789·0 | 789·8 | 790·5 |
| 1275 | 784·3 | 785·1 | 785·9 | 786·7 | 787·4 |
| 1280 | 781·2 | 782·0 | 782·8 | 783·6 | 784·4 |
| 1285 | 778·2 | 779·0 | 779·8 | 780·5 | 781·3 |
| 1290 | 775·2 | 776·0 | 776·7 | 777·5 | 778·3 |
| 1295 | 772·2 | 773·0 | 773·7 | 774·5 | 775·3 |
| 1300 | 769·2 | 770·0 | 770·8 | 771·5 | 772·3 |
| 1305 | 766·3 | 767·0 | 767·8 | 768·6 | 769·3 |
| 1310 | 763·4 | 764·1 | 764·9 | 765·6 | 766·4 |
| 1315 | 760·5 | 761·2 | 762·0 | 762·7 | 763·5 |
| 1320 | 757·6 | 758·3 | 759·1 | 759·8 | 760·6 |
| 1325 | 754·7 | 755·5 | 756·2 | 757·0 | 757·7 |
| 1330 | 751·9 | 752·6 | 753·4 | 754·1 | 754·9 |
| 1335 | 749·1 | 749·8 | 750·6 | 751·3 | 752·1 |
| 1340 | 746·3 | 747·0 | 747·8 | 748·5 | 749·2 |
| 1345 | 743·5 | 744·2 | 745·0 | 745·7 | 746·5 |
| 1350 | 740·7 | 741·5 | 742·2 | 743·0 | 743·7 |
| 1355 | 738·0 | 738·7 | 739·5 | 740·2 | 741·0 |
| 1360 | 735·3 | 736·0 | 736·8 | 737·5 | 738·2 |
| 1365 | 732·6 | 733·3 | 734·1 | 734·8 | 735·5 |
| 1370 | 729·9 | 730·7 | 731·4 | 732·1 | 732·8 |
| 1375 | 727·3 | 728·0 | 728·7 | 729·4 | 730·2 |
| 1380 | 724·6 | 725·4 | 726·1 | 726·8 | 727·5 |
| 1385 | 722·0 | 722·7 | 723·5 | 724·2 | 724·9 |
| 1390 | 719·4 | 720·1 | 720·9 | 721·6 | 722·3 |
| 1395 | 716·8 | 717·6 | 718·3 | 719·0 | 719·7 |
| 1400 | 714·3 | 715·0 | 715·7 | 716·4 | 717·1 |

SALT.—*continued.*

| 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|
| 844·5 | 845·4 | 846·2 | 847·1 | 847·9 | 848·7 |
| 841·0 | 841·8 | 842·7 | 843·5 | 844·3 | 845·2 |
| 837·5 | 838·3 | 839·2 | 840·0 | 840·8 | 841·7 |
| 834·0 | 834·8 | 835·7 | 836·5 | 837·3 | 838·2 |
| 830·6 | 831·4 | 832·2 | 833·1 | 833·9 | 834·7 |
| 827·2 | 828·0 | 828·8 | 829·6 | 830·4 | 831·3 |
| 823·8 | 824·6 | 825·4 | 826·2 | 827·0 | 827·9 |
| 820·4 | 821·2 | 822·0 | 822·9 | 823·7 | 824·5 |
| 817·1 | 817·9 | 818·7 | 819·5 | 820·3 | 821·1 |
| 813·8 | 814·6 | 815·4 | 816·2 | 817·0 | 817·8 |
| 810·5 | 811·3 | 812·1 | 812·9 | 813·7 | 814·5 |
| 807·2 | 808·0 | 808·8 | 809·6 | 810·4 | 811·2 |
| 804·0 | 804·8 | 805·6 | 806·4 | 807·2 | 808·0 |
| 800·8 | 801·6 | 802·4 | 803·2 | 804·0 | 804·8 |
| 797·6 | 798·4 | 799·2 | 800·0 | 800·8 | 801·6 |
| 794·5 | 795·3 | 796·0 | 796·8 | 797·6 | 798·4 |
| 791·3 | 792·1 | 792·9 | 793·7 | 794·5 | 795·3 |
| 788·2 | 789·0 | 789·8 | 790·6 | 791·4 | 792·2 |
| 785·2 | 786·0 | 786·7 | 787·5 | 788·3 | 789·1 |
| 782·1 | 782·9 | 783·7 | 784·4 | 785·2 | 786·0 |
| 779·1 | 779·8 | 780·6 | 781·4 | 782·2 | 782·9 |
| 776·1 | 776·8 | 777·6 | 778·4 | 779·1 | 779·9 |
| 773·1 | 773·8 | 774·6 | 775·4 | 776·1 | 776·9 |
| 770·1 | 770·9 | 771·6 | 772·4 | 773·2 | 773·9 |
| 767·2 | 767·9 | 768·7 | 769·5 | 770·2 | 771·0 |
| 764·3 | 765·0 | 765·8 | 766·5 | 767·3 | 768·1 |
| 761·4 | 762·1 | 762·9 | 763·6 | 764·4 | 765·2 |
| 758·5 | 759·2 | 760·0 | 760·7 | 761·5 | 762·3 |
| 755·6 | 756·4 | 757·1 | 757·9 | 758·6 | 759·4 |
| 752·8 | 753·6 | 754·3 | 755·1 | 755·8 | 756·6 |
| 750·0 | 750·7 | 751·5 | 752·2 | 753·0 | 753·7 |
| 747·2 | 748·0 | 748·7 | 749·4 | 750·2 | 750·9 |
| 744·4 | 745·2 | 745·9 | 746·7 | 747·4 | 748·1 |
| 741·7 | 742·4 | 743·2 | 743·9 | 744·6 | 745·4 |
| 739·0 | 739·7 | 740·4 | 741·2 | 741·9 | 742·6 |
| 736·3 | 737·0 | 737·7 | 738·5 | 739·2 | 739·9 |
| 733·6 | 734·3 | 735·0 | 735·8 | 736·5 | 737·2 |
| 730·9 | 731·6 | 732·4 | 733·2 | 733·8 | 734·5 |
| 728·3 | 729·0 | 729·7 | 730·4 | 731·2 | 731·9 |
| 725·6 | 726·3 | 727·1 | 727·8 | 728·5 | 729·2 |
| 723·0 | 723·7 | 724·5 | 725·2 | 725·9 | 726·6 |
| 720·4 | 721·1 | 721·9 | 722·6 | 723·3 | 724·0 |
| 717·9 | 718·6 | 719·3 | 720·0 | 720·7 | 721·4 |

COMMON

| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
|---|---|---|---|---|---|
| 1405 | 711·7 | 712·5 | 713·2 | 713·9 | 714·6 |
| 1410 | 709·2 | 709·9 | 710·6 | 711·3 | 712·1 |
| 1415 | 706·7 | 707·4 | 708·1 | 708·8 | 709·5 |
| 1420 | 704·2 | 704·9 | 705·6 | 706·3 | 707·0 |
| 1425 | 701·8 | 702·5 | 703·2 | 703·9 | 704·6 |
| 1430 | 699·3 | 700·0 | 700·7 | 701·4 | 702·1 |
| 1435 | 696·9 | 697·6 | 698·3 | 698·9 | 699·6 |
| 1440 | 694·4 | 695·1 | 695·8 | 696·5 | 697·2 |
| 1445 | 692·0 | 692·7 | 693·4 | 694·1 | 694·8 |
| 1450 | 689·7 | 690·3 | 691·0 | 691·7 | 692·4 |
| 1455 | 687·3 | 688·0 | 688·7 | 689·3 | 690·0 |
| 1460 | 684·9 | 685·6 | 686·3 | 687·0 | 687·7 |
| 1465 | 682·6 | 683·3 | 684·0 | 684·6 | 685·3 |
| 1470 | 680·3 | 680·9 | 681·6 | 682·3 | 683·0 |
| 1475 | 678·0 | 678·6 | 679·3 | 680·0 | 680·7 |
| 1480 | 675·7 | 676·3 | 677·0 | 677·7 | 678·4 |
| 1485 | 673·4 | 674·1 | 674·7 | 675·4 | 676·1 |
| 1490 | 671·1 | 671·8 | 672·5 | 673·1 | 673·8 |
| 1495 | 668·9 | 669·6 | 670·2 | 670·9 | 671·6 |
| 1500 | 666·7 | 667·3 | 668·0 | 668·7 | 669·3 |
| 1505 | 664·5 | 665·1 | 665·8 | 666·4 | 667·1 |
| 1510 | 662·3 | 662·9 | 663·6 | 664·2 | 664·9 |
| 1515 | 660·1 | 660·7 | 661·4 | 662·0 | 662·7 |
| 1520 | 657·9 | 658·5 | 659·2 | 659·9 | 660·5 |
| 1525 | 655·7 | 656·4 | 657·0 | 657·7 | 658·4 |
| 1530 | 653·6 | 654·2 | 654·9 | 655·6 | 656·2 |
| 1535 | 651·5 | 652·1 | 652·8 | 653·4 | 654·1 |
| 1540 | 649·4 | 650·0 | 650·6 | 651·3 | 651·9 |
| 1545 | 647·2 | 647·9 | 648·5 | 649·2 | 649·8 |
| 1550 | 645·2 | 645·8 | 646·4 | 647·1 | 647·7 |
| 1555 | 643·1 | 643·7 | 644·4 | 645·0 | 645·7 |
| 1560 | 641·0 | 641·7 | 642·3 | 642·9 | 643·6 |
| 1565 | 639·0 | 639·6 | 640·3 | 640·9 | 641·5 |
| 1570 | 636·9 | 637·6 | 638·2 | 638·8 | 639·5 |
| 1575 | 634·9 | 635·6 | 636·2 | 636·8 | 637·5 |
| 1580 | 632·9 | 633·5 | 634·2 | 634·8 | 635·4 |
| 1585 | 630·9 | 631·5 | 632·2 | 632·8 | 633·4 |
| 1590 | 628·9 | 629·6 | 630·2 | 630·8 | 631·4 |
| 1595 | 627·0 | 627·6 | 628·2 | 628·8 | 629·5 |
| 1600 | 625·0 | 625·6 | 626·2 | 626·9 | 627·5 |
| 1605 | 623·1 | 623·7 | 624·3 | 624·9 | 625·5 |
| 1610 | 621·1 | 621·7 | 622·4 | 623·0 | 623·6 |
| 1615 | 619·2 | 619·8 | 620·4 | 621·0 | 621·7 |

SALT—*continued.*

| 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|
| 715·3 | 716·0 | 716·7 | 717·4 | 718·1 | 718·9 |
| 712·8 | 713·5 | 714·2 | 714·9 | 715·6 | 716·3 |
| 710·2 | 710·9 | 711·7 | 712·4 | 713·1 | 713·8 |
| 707·7 | 708·4 | 709·2 | 709·9 | 710·6 | 711·3 |
| 705·3 | 706·0 | 706·7 | 707·4 | 708·1 | 708·8 |
| 702·8 | 703·5 | 704·2 | 704·9 | 705·6 | 706·3 |
| 700·3 | 701·0 | 701·7 | 702·4 | 703·1 | 703·8 |
| 697·9 | 698·6 | 699·3 | 700·0 | 700·7 | 701·4 |
| 695·5 | 696·2 | 696·9 | 697·6 | 698·3 | 699·0 |
| 693·1 | 693·8 | 694·5 | 695·2 | 695·9 | 696·6 |
| 690·7 | 691·4 | 692·1 | 692·8 | 693·5 | 694·2 |
| 688·4 | 689·0 | 689·7 | 690·4 | 691·1 | 691·8 |
| 686·0 | 686·7 | 687·4 | 688·0 | 688·7 | 689·4 |
| 683·7 | 684·3 | 685·0 | 685·7 | 686·4 | 687·1 |
| 681·4 | 682·0 | 682·7 | 683·4 | 684·1 | 684·7 |
| 679·1 | 679·7 | 680·4 | 681·1 | 681·8 | 682·4 |
| 676·8 | 677·4 | 678·1 | 678·8 | 679·5 | 680·1 |
| 674·5 | 675·2 | 675·8 | 676·5 | 677·2 | 677·8 |
| 672·2 | 672·9 | 673·6 | 674·2 | 674·9 | 675·6 |
| 670·0 | 670·7 | 671·3 | 672·0 | 672·7 | 673·3 |
| 667·8 | 668·4 | 669·1 | 669·8 | 670·4 | 671·1 |
| 665·6 | 666·2 | 666·9 | 667·5 | 668·2 | 668·9 |
| 663·4 | 664·0 | 664·7 | 665·3 | 666·0 | 666·7 |
| 661·2 | 661·8 | 662·5 | 663·2 | 663·8 | 664·5 |
| 659·0 | 659·7 | 660·3 | 661·0 | 661·6 | 662·3 |
| 656·9 | 657·5 | 658·2 | 658·8 | 659·5 | 660·1 |
| 654·7 | 655·4 | 656·0 | 656·7 | 657·3 | 658·0 |
| 652·6 | 653·2 | 653·9 | 654·5 | 655·2 | 655·8 |
| 650·5 | 651·1 | 651·8 | 652·4 | 653·1 | 653·7 |
| 648·4 | 649·0 | 649·7 | 650·3 | 651·0 | 651·6 |
| 646·3 | 646·9 | 647·6 | 648·2 | 648·9 | 649·5 |
| 644·2 | 644·9 | 645·5 | 646·1 | 646·8 | 647·4 |
| 642·2 | 642·8 | 643·4 | 644·1 | 644·7 | 645·4 |
| 640·1 | 640·8 | 641·4 | 642·0 | 642·7 | 643·3 |
| 638·1 | 638·7 | 639·4 | 640·0 | 640·6 | 641·3 |
| 636·1 | 636·7 | 637·3 | 638·0 | 638·6 | 639·2 |
| 634·1 | 634·7 | 635·3 | 636·0 | 636·6 | 637·2 |
| 632·1 | 632·7 | 633·3 | 634·0 | 634·6 | 635·2 |
| 630·1 | 630·7 | 631·3 | 632·0 | 632·6 | 633·2 |
| 628·1 | 628·7 | 629·4 | 630·0 | 630·6 | 631·2 |
| 626·2 | 626·8 | 627·4 | 628·0 | 628·7 | 629·3 |
| 624·2 | 624·8 | 625·5 | 626·1 | 626·7 | 627·3 |
| 622·3 | 622·9 | 623·5 | 624·1 | 624·8 | 625·4 |

COMMON

| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
|---|---|---|---|---|---|
| 1620 | 617·3 | 617·9 | 618·5 | 619·1 | 619·7 |
| 1625 | 615·4 | 616·0 | 616·6 | 617·2 | 617·8 |
| 1630 | 613·5 | 614·1 | 614·7 | 615·3 | 615·9 |
| 1635 | 611·6 | 612·2 | 612·8 | 613·5 | 614·1 |
| 1640 | 609·8 | 610·4 | 611·0 | 611·6 | 612·2 |
| 1645 | 607·9 | 608·5 | 609·1 | 609·7 | 610·3 |
| 1650 | 606·1 | 606·7 | 607·3 | 607·9 | 608·5 |
| 1655 | 604·2 | 604·8 | 605·4 | 606·0 | 606·6 |
| 1660 | 602·4 | 603·0 | 603·6 | 604·2 | 604·8 |
| 1665 | 600·6 | 601·2 | 601·8 | 602·4 | 603·0 |
| 1670 | 598·8 | 699·4 | 600·0 | 600·6 | 601·2 |
| 1675 | 597·0 | 597·6 | 598·2 | 598·8 | 599·4 |
| 1680 | 595·2 | 595·8 | 596·4 | 597·0 | 597·6 |
| 1685 | 593·5 | 594·1 | 594·7 | 595·2 | 595·8 |
| 1690 | 591·7 | 592·3 | 592·9 | 593·5 | 594·1 |
| 1695 | 590·0 | 590·6 | 591·1 | 591·7 | 592·3 |
| 1700 | 588·2 | 588·8 | 589·4 | 590·0 | 590·6 |
| 1705 | 586·5 | 587·1 | 587·7 | 588·3 | 588·9 |
| 1710 | 584·8 | 585·4 | 586·0 | 586·5 | 587·1 |
| 1715 | 583·1 | 583·7 | 584·3 | 584·8 | 585·4 |
| 1720 | 581·4 | 582·0 | 582·6 | 583·1 | 583·7 |
| 1725 | 579·7 | 580·3 | 580·9 | 581·4 | 582·0 |
| 1730 | 578·0 | 578·6 | 579·2 | 579·8 | 580·3 |
| 1735 | 576·4 | 576·9 | 577·5 | 578·1 | 578·7 |
| 1740 | 574·7 | 575·3 | 575·9 | 576·4 | 577·0 |
| 1745 | 573·1 | 573·6 | 574·2 | 574·8 | 575·4 |
| 1750 | 571·4 | 572·0 | 572·6 | 573·1 | 573·7 |
| 1755 | 569·8 | 570·4 | 570·9 | 571·5 | 572·1 |
| 1760 | 568·2 | 568·7 | 569·3 | 569·9 | 570·4 |
| 1765 | 566·6 | 567·1 | 567·7 | 568·3 | 568·8 |
| 1770 | 565·0 | 565·5 | 566·1 | 566·7 | 567·2 |
| 1775 | 563·4 | 563·9 | 564·5 | 565·1 | 565·6 |
| 1780 | 561·8 | 562·4 | 562·9 | 563·5 | 564·0 |
| 1785 | 560·2 | 560·8 | 561·3 | 561·9 | 562·5 |
| 1790 | 558·7 | 559·2 | 559·8 | 560·3 | 560·9 |
| 1795 | 557·1 | 557·7 | 558·2 | 558·8 | 559·3 |
| 1800 | 555·6 | 556·1 | 556·7 | 557·2 | 557·8 |
| 1805 | 554·0 | 554·6 | 555·1 | 555·7 | 556·2 |
| 1810 | 552·5 | 553·0 | 553·6 | 554·1 | 554·7 |
| 1815 | 551·0 | 551·5 | 552·1 | 552·6 | 553·2 |
| 1820 | 549·4 | 550·0 | 550·5 | 551·1 | 551·6 |
| 1825 | 547·9 | 548·5 | 549·0 | 549·6 | 550·1 |
| 1830 | 546·4 | 547·0 | 547·5 | 548·1 | 548·6 |

SALT—*continued.*

| 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|
| 620·4 | 621·0 | 621·6 | 622·2 | 622·8 | 623·5 |
| 618·5 | 619·1 | 619·7 | 620·3 | 620·9 | 621·5 |
| 616·6 | 617·2 | 617·8 | 618·4 | 619·0 | 619·6 |
| 614·7 | 615·3 | 615·9 | 616·5 | 617·1 | 617·7 |
| 612·8 | 613·4 | 614·0 | 614·6 | 615·2 | 615·8 |
| 610·9 | 611·5 | 612·2 | 612·8 | 613·4 | 614·0 |
| 609·1 | 609·7 | 610·3 | 610·9 | 611·5 | 612·1 |
| 607·2 | 607·8 | 608·5 | 609·1 | 609·7 | 610·3 |
| 605·4 | 606·0 | 606·6 | 607·2 | 607·8 | 608·4 |
| 603·6 | 604·2 | 604·8 | 605·4 | 606·0 | 606·6 |
| 601·8 | 602·4 | 603·0 | 603·6 | 604·2 | 604·8 |
| 600·0 | 600·6 | 601·2 | 601·8 | 602·4 | 603·0 |
| 598·2 | 598·8 | 599·4 | 600·0 | 600·6 | 601·2 |
| 596·4 | 597·0 | 597·6 | 598·2 | 598·8 | 599·4 |
| 594·7 | 595·3 | 595·9 | 596·4 | 597·0 | 597·6 |
| 592·9 | 593·5 | 594·1 | 594·7 | 595·3 | 595·9 |
| 591·2 | 591·8 | 592·3 | 592·9 | 593·5 | 594·1 |
| 589·4 | 590·0 | 590·6 | 591·2 | 591·8 | 592·4 |
| 587·7 | 588·3 | 588·9 | 589·5 | 590·1 | 590·6 |
| 586·0 | 586·6 | 587·2 | 587·8 | 588·3 | 588·9 |
| 584·3 | 584·9 | 585·5 | 586·0 | 586·6 | 587·2 |
| 582·6 | 583·2 | 583·8 | 584·3 | 584·9 | 585·5 |
| 580·9 | 581·5 | 582·1 | 582·7 | 583·2 | 583·8 |
| 579·2 | 579·8 | 580·4 | 581·0 | 581·6 | 582·1 |
| 577·6 | 578·2 | 578·7 | 579·3 | 579·9 | 580·5 |
| 575·9 | 576·5 | 577·1 | 577·6 | 578·2 | 578·8 |
| 574·3 | 574·9 | 575·4 | 576·0 | 576·6 | 577·1 |
| 572·6 | 573·2 | 573·8 | 574·4 | 574·9 | 575·5 |
| 571·0 | 571·6 | 572·2 | 572·7 | 573·3 | 573·9 |
| 569·4 | 570·0 | 570·5 | 571·1 | 571·7 | 572·2 |
| 567·8 | 568·4 | 568·9 | 569·5 | 570·1 | 570·6 |
| 566·2 | 566·8 | 567·3 | 567·9 | 568·4 | 569·0 |
| 564·6 | 565·2 | 565·7 | 566·3 | 566·8 | 567·4 |
| 563·0 | 563·6 | 564·1 | 564·7 | 565·3 | 565·8 |
| 561·4 | 562·0 | 562·6 | 563·1 | 563·7 | 564·2 |
| 559·9 | 560·4 | 561·0 | 561·6 | 562·1 | 562·7 |
| 558·3 | 558·9 | 559·4 | 560·0 | 560·6 | 561·1 |
| 556·8 | 557·3 | 557·9 | 558·4 | 559·0 | 559·6 |
| 555·2 | 555·8 | 556·3 | 556·9 | 557·5 | 558·0 |
| 553·7 | 554·3 | 554·8 | 555·4 | 555·9 | 556·5 |
| 552·2 | 552·7 | 553·3 | 553·8 | 554·4 | 554·9 |
| 550·7 | 551·2 | 551·8 | 552·3 | 552·9 | 553·4 |
| 549·2 | 549·7 | 550·3 | 550·8 | 551·4 | 551·9 |

COMMON

| Weight of Assay in Milligrs. | 0. | 1. | 2. | 3. | 4. |
|---|---|---|---|---|---|
| 1835 | 545·0 | 545·5 | 546·0 | 546·6 | 547·1 |
| 1840 | 543·5 | 544·0 | 544·6 | 545·1 | 545·6 |
| 1845 | 542·0 | 542·5 | 543·1 | 543·6 | 544·2 |
| 1850 | 540·5 | 541·1 | 541·6 | 542·2 | 542·7 |
| 1855 | 539·1 | 539·6 | 540·2 | 540·7 | 541·2 |
| 1860 | 537·6 | 538·2 | 538·7 | 539·2 | 539·8 |
| 1865 | 536·2 | 536·7 | 537·3 | 537·8 | 538·3 |
| 1870 | 534·8 | 535·3 | 535·8 | 536·4 | 536·9 |
| 1875 | 533·3 | 533·9 | 534·4 | 534·9 | 535·5 |
| 1880 | 531·9 | 532·4 | 533·0 | 533·5 | 534·0 |
| 1885 | 530·5 | 531·0 | 531·6 | 532·1 | 532·6 |
| 1890 | 529·1 | 529·6 | 530·2 | 530·7 | 531·2 |
| 1895 | 527·7 | 528·2 | 528·8 | 529·3 | 529·8 |
| 1900 | 526·3 | 526·8 | 527·4 | 527·9 | 528·4 |
| 1905 | 524·9 | 525·4 | 526·0 | 526·5 | 527·0 |
| 1910 | 523·6 | 524·1 | 524·6 | 525·1 | 525·6 |
| 1915 | 522·2 | 522·7 | 523·2 | 523·8 | 524·3 |
| 1920 | 520·8 | 521·3 | 521·9 | 522·4 | 522·9 |
| 1925 | 519·5 | 520·0 | 520·5 | 521·0 | 521·6 |
| 1930 | 518·1 | 518·6 | 519·2 | 519·7 | 520·2 |
| 1935 | 516·8 | 517·3 | 517·8 | 518·3 | 518·9 |
| 1940 | 515·5 | 516·0 | 516·5 | 517·0 | 517·5 |
| 1945 | 514·1 | 514·6 | 515·2 | 515·7 | 516·2 |
| 1950 | 512·8 | 513·3 | 513·8 | 514·4 | 514·9 |
| 1955 | 511·5 | 512·0 | 512·5 | 513·0 | 513·5 |
| 1960 | 510·2 | 510·7 | 511·2 | 511·7 | 512·2 |
| 1965 | 508·9 | 509·4 | 509·9 | 510·4 | 510·9 |
| 1970 | 507·6 | 508·1 | 508·6 | 509·1 | 509·6 |
| 1975 | 506·3 | 506·8 | 507·3 | 507·8 | 508·3 |
| 1980 | 505·0 | 505·6 | 506·1 | 506·6 | 507·1 |
| 1985 | 503·8 | 504·3 | 504·8 | 505·3 | 505·8 |
| 1990 | 502·5 | 503·0 | 503·5 | 504·0 | 504·5 |
| 1995 | 501·3 | 501·8 | 502·3 | 502·8 | 503·3 |
| 2000 | 500·0 | 500·5 | 501·0 | 501·5 | 502·0 |

SALT—*continued.*

| 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|
| 547·7 | 548·2 | 548·8 | 549·3 | 549·9 | 550·4 |
| 546·2 | 546·7 | 547·3 | 547·8 | 548·4 | 548·9 |
| 544·7 | 545·3 | 545·8 | 546·3 | 546·9 | 547·4 |
| 543·2 | 543·8 | 544·3 | 544·9 | 545·4 | 545·9 |
| 541·8 | 542·3 | 542·9 | 543·4 | 543·9 | 544·5 |
| 540·3 | 540·9 | 541·4 | 541·9 | 542·5 | 543·0 |
| 538·9 | 539·4 | 539·9 | 540·5 | 541·0 | 541·5 |
| 537·4 | 538·0 | 538·5 | 539·0 | 539·6 | 540·1 |
| 536·0 | 536·5 | 537·1 | 537·6 | 538·1 | 538·7 |
| 534·6 | 535·1 | 535·6 | 536·2 | 536·7 | 537·2 |
| 533·2 | 533·7 | 534·2 | 534·7 | 535·3 | 535·8 |
| 531·7 | 532·3 | 532·8 | 533·3 | 533·9 | 534·4 |
| 530·3 | 530·9 | 531·4 | 531·9 | 532·4 | 533·0 |
| 528·9 | 529·5 | 530·0 | 530·5 | 531·0 | 531·6 |
| 527·6 | 528·1 | 528·6 | 529·1 | 529·7 | 530·2 |
| 526·2 | 526·7 | 527·2 | 527·7 | 528·3 | 528·8 |
| 524·8 | 525·3 | 525·8 | 526·4 | 526·9 | 527·4 |
| 523·4 | 524·0 | 524·5 | 525·0 | 525·5 | 526·0 |
| 522·1 | 522·6 | 523·1 | 523·6 | 524·2 | 524·7 |
| 520·7 | 521·2 | 521·8 | 522·3 | 522·8 | 523·3 |
| 519·4 | 519·9 | 520·4 | 520·9 | 521·4 | 522·0 |
| 518·0 | 518·6 | 519·1 | 519·6 | 520·1 | 520·6 |
| 516·7 | 517·2 | 517·7 | 518·2 | 518·8 | 519·3 |
| 515·4 | 515·9 | 516·4 | 516·9 | 517·4 | 517·9 |
| 514·1 | 514·6 | 515·1 | 515·6 | 516·1 | 516·6 |
| 512·8 | 513·3 | 513·8 | 514·3 | 514·8 | 515·3 |
| 511·4 | 512·0 | 512·5 | 513·0 | 513·5 | 514·0 |
| 510·1 | 510·7 | 511·2 | 511·7 | 512·2 | 512·7 |
| 508·9 | 509·4 | 509·9 | 510·4 | 510·9 | 511·4 |
| 507·6 | 508·1 | 508·6 | 509·1 | 509·6 | 510·1 |
| 506·3 | 506·8 | 507·3 | 507·8 | 508·3 | 508·8 |
| 505·0 | 505·5 | 506·0 | 506·5 | 507·0 | 507·5 |
| 503·8 | 504·3 | 504·8 | 505·3 | 505·8 | 506·3 |
| 502·5 | 503·0 | 503·5 | 504·0 | 504·5 | 505·0 |

## APPLICATION.

### *Assay of Pure, or nearly Pure, Silver, the Temperature of the Normal Solution of Salt being that at which it was standardised.*

*First Example.*—Let the ingot of silver have an approximative standard of from 995 to 1000 thousandths. Take one gramme; dissolve it in ten grammes of nitric acid, in the bottle, fig. 89. Then pour into the bottle an exact measure of the normal solution of salt, and brighten by agitation. The silver not being supposed to be quite pure, the standard is not further sought for by the decime solution of salt, but that of nitrate of silver is employed.

One thousandth of this latter solution is poured into the bottle; it becomes cloudy, and is well agitated. A second and a third thousandth also give a precipitate, but not so a fourth. From these data the following is the method of ascertaining the standard of the alloy:—

The last thousandth of the decime solution of silver, having produced no cloudiness, is not to be counted. The third was necessary, but only partially so; consequently the number of thousandths of silver necessary to decompose the excess of salt is more than 2 and less than 3; in other words, it is equal to the mean, 2½; but since 2½ thousandths of silver have been required to complete the precipitation of salt representing 1000 thousandths of silver, it is evident that the silver submitted to assay contained 2½ thousandths of alloy, and that its standard, to within nearly half a thousandth, is but 997½.

If it be considered necessary to arrive nearer the true standard, the following proofs must be employed:—Pour into the solution 1½ thousandths of salt, which will decompose a like number of thousandths of silver.* After due agitation, add half a thousandth of nitrate of silver. Supposing a cloudiness is produced, no further addition must be made; for it is already known that above the third thousandth no precipitate is formed in the liquid by nitrate of silver, and consequently only half of the last half thousandth must be calculated, as only a portion of it was necessary. From which, the entire number of thousandths of nitrate of silver being 4¼, and those of salt 1½, there remains 2¾ for the number of thousandths of nitrate of silver added to the normal solution; and consequently the standard of the alloy is 1000−2¾=997¼. If, on the other hand, the last half thousandth of nitrate of silver had produced no cloudiness, it would not have to be reckoned, and only half of the preceding half thousandth would have been taken. Thus from the 4 thousandths of nitrate of silver employed a quarter of a thousandth is deducted; and from the difference, 3¾, is yet deducted 1½ of salt, the final remainder being 2¼ thousandths of nitrate of silver which have been added to the normal solution: the standard of the alloy would be 1000−2¼=997¾.

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* It has already been stated how a thousandth of the decime solution may be subdivided by the number of drops furnished by the pipette. If, for instance, it contains 20 drops, 10 will give the half, 5 the quarter, &c. Half a thousandth may also be obtained by diluting the solution with its volume of water, and using a whole pipetteful. This latter plan has been found the best in practice.

Although the above-described operation is very simple, yet it is desirable, in order to avoid all confusion, to note in writing such thousandths of salt or nitrate of silver as are added. The thousandths of salt indicating an increase of standard should be preceded by the sign +; and the thousandths of nitrate of silver announcing a diminution of standard, by the sign −.

*Second Example.*—Suppose the ingot has a presumed standard of 895 thousandths, and the temperature of the normal solution supposed invariable.

Find in the table of standards (Salt Table), first column, that which approaches the nearest to 895; it will be found to be 896·9, corresponding to the weight of 1115 milligrammes. This weight of the alloy is taken and dissolved in nitric acid, a measure of normal solution of salt added, and the whole well agitated. The operator is, however, doubtful whether the assay must be proceeded with by the decime salt solution, or the nitrate of silver decime solution. If the former produces a precipitate, it is gone on with;

but if it does not precipitate, that already added is decomposed by a similar addition of the second, and the solution rendered bright by agitation. A starting-point has now been arrived at for the continuance of the assay, for it is known that the nitrate of silver solution must be employed.

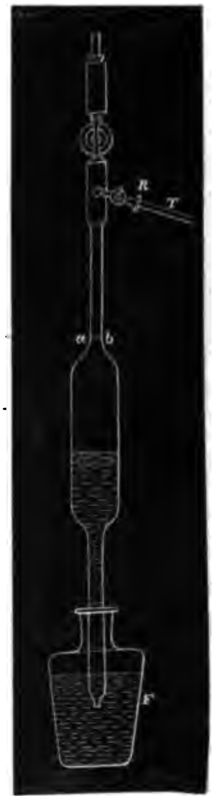
Suppose, then, that the alloy, after the addition of the measure of normal solution, yet gives a precipitate with the decime solution of salt. The first 5 thousandths produce a precipitate, but not the sixth, which consequently is not counted. The fifth has only been partially required, so that it is more than 4 thousandths, and less than 5, or the mean, 4½, is the quantity required to entirely precipitate the excess of silver in the alloy submitted to assay. But by neglecting at first the fraction 0·5, seek in the Salt Table of Standards the number found on the longitudinal line of the weight 1115, under column 4; it is 900·4, and on adding 0·5 to this number, we have 900·9, or 901, for the required standard.

Supposing, however, that the same alloy, after the addition of the normal measure of salt, gives a precipitate with nitrate of silver, and that the 3 first thousandths produce a cloudiness, but not the fourth. The number of thousandths of nitrate of silver really necessary for complete precipitation will be very nearly 2½. To ascertain the real standard of the alloy of which 1115 thousandths were supposed to contain about 1000 thousandths of silver, take the number found in the horizontal line 1115, and in the column 2 of the Nitrate of Silver Table. This number, which is 895·1, diminished by the fraction 0·5, gives 894·6 for the standard of the alloy to within half a thousandth.

*Third Example.*—The actual temperature of the normal solution of salt being 18° when it was standardised at 15°.

The ingot of silver submitted to assay has an approximative standard of 795 thousandths. Find in the Salt Table of Standards, first column, that which is nearest to it; it is 793·7, corresponding to the weight 1260. This weight of the alloy is taken, and the operation proceeded with as already described. Supposing it had required 6·5 thousandths of salt to precipitate the whole of the silver contained in the alloy to within half a thousandth, the required standard, without correction for temperature, will be 798·4+0·4=798·8. But, making this correction, recourse must be had to the table, page 522, column 15: the number 0·3, which will be found in the horizontal line 18 and the column 15, possesses the − sign; consequently it must be deducted from 798·8, and the remainder, 798·5, will be the standard weight. If the temperature of the solution, instead of being 3° higher than at the time it was standardised, was 3° lower, or 12°, the correction must be added, and would be equal to +0·2. The standard of the alloy would consequently be 798·8+0·2=799.

Fig. 104.



### *Graduation of the Normal Solution of Salt, the Temperature being different to that at which it is wished to be graduated.*

Two equally ready processes can be employed. The one consists in reducing the temperature of the solution to the desired degree before standardising; the other, in determining its standard without regard to the temperature of the solution, and then correcting its influence by the aid of the tables of correction already given.

*First Process.*—Place the liquid to be graduated in a bottle, *F*, fig. 104. Introduce a thermometer, and heat to a determinate degree, say 20° for instance. This done, place the jet of the pipette in the bottle: raise the liquid by aspiration by means of the conical tube, *T*, fig. 98, which is adapted to the opening of the air-cock, *R*. As soon as the liquid is raised a little above the mark *a b*, which determines the capacity of the pipette, close the stopcock, and complete the measurement as usual. This same means of filling the pipette by aspiration may be employed to fill it either with caustic alkali or nitric acid, as the case may be, to cleanse it instead of taking it to pieces.

*Second Method.*—The solution of salt being supposed at a temperature of 16°, and it is desired to graduate it at that of 20°. Proceed with the standardising without regard to temperature; but when it is obtained in each trial assay, it is necessary to make the correction required by the temperature.

If, for example, in an approximative assay the standard of the solution was expressed by 1001·5, this standard would not only be too weak by 1·5 thousandth, but, according to the table of temperatures, by yet another 0·5, for the solution is weakened by this quantity by passing from 16° to 20°. The standard, if taken at this last temperature, would be too low by 2 thousandths, and must consequently be corrected.

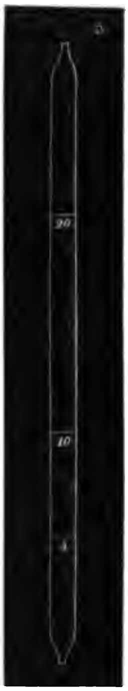
If, on the other hand, the standard of the solution were too high instead of too low, and expressed by 998·5 at the temperature of 16°; at that of 20°, the solution being weakened by 0·5, the standard would only be but one thousandth too high, and it must be corrected by that quantity.

### *Approximative Determination of the Standard of an Unknown Alloy.*

It has always been supposed, in the experiments already detailed, that the approximative standard of the alloy submitted to assay was known: and this, indeed, is nearly always the case. If, however, this be unknown, two means are available for obtaining the necessary knowledge. A decigramme of the alloy is cupelled with one gramme of lead; or, if it be desirable not to use the cupel, it may be ascertained by the humid method, in the following manner:—

The assayer supposes the standard of the alloy known to about a twentieth, and it can always be found nearer than that by touch, density, &c. A weight relative to its supposed standard is taken, and its standard sought by adding the decime liquid by 10 thousandths at a time, by means of pipettes of this capacity (see fig. 105). The term of complete precipitation is soon passed, and the standard of the alloy to about 5 thousandths is thus ascertained. The approximate standard to 2½ thousandths may be obtained by adding only 5 thousandths of solution at a time.

FIG. 105.



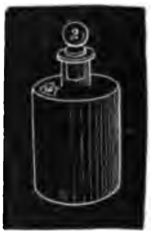
Suppose the alloy 840 thousandths. Take the weight 1190, corresponding to this standard, and proceed as in an ordinary assay, adding each time, for example, a pipette of 10 thousandths of salt solution. It is found the fifth pipette gives no precipitate, and consequently the number of thousands of salt for the precipitate of the silver to within 5 thousandths is 35. The 1199 of alloy will therefore contain $1000+35=1035$ of silver; and the approximative standard will be obtained by the proportion—

$$1120 : 1035 :: 1000 : x=869{\cdot}7.$$

### *Modes of Abridging Manipulation.*

In the statement already given of the mode of conducting the assay by the wet method, only such instructions have been given as were necessary for its full comprehension, and everything that might call away or fatigue the attention has been omitted. Nevertheless, here it will be convenient to describe some methods of abridging the necessary manipulations, supposing that ten, or at least five, assays are made at once.

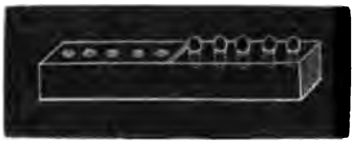
FIG. 106.



*Bottles.*—It is necessary to have these all, as nearly as possible, of the same height and diameter. They are marked progressively on the shoulder, as are also their stoppers (fig. 106), thus—1, 2, 3, 4 &c. They are taken successively by tens, in the natural order.

The stoppers are placed on a support, numbered in the same manner (fig. 107). The support is pierced with ten holes, distinguished in precedence by a mark between the fifth and sixth.

Fig. 107.



*Stand.*—Each ten flasks are in turn placed in a case or stand of japanned tin-plate (fig. 108), having ten compartments numbered from 1 to 10. Each of these compartments is cut out anteriorly to about half their length, so as to allow the numbers of the bottles to be seen. The same stand serves for all the series, by making the same units correspond: thus No. 23 of the third series is placed in stand No. 3, &c. When each flask is charged with the alloy, about 10 grammes of nitric acid, 40° C., are measured by a pipette (fig. 93) introduced into the bottles by means of a funnel with a large neck (fig. 109). The whole are then exposed to the heat of a water-bath, to facilitate the solution of the alloy.

Fig. 108.

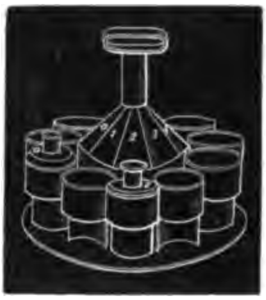


Fig. 109.

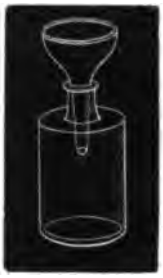
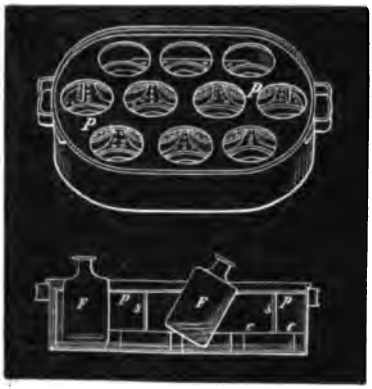


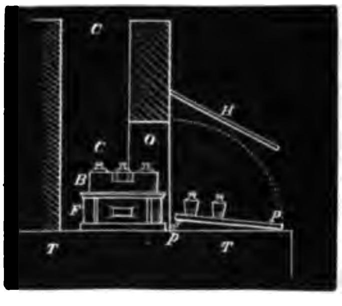
Fig. 110.



*Water-bath.*—This is an oblong tin-plate vessel, calculated to receive 10 bottles (fig. 110). It has a movable double bottom, pierced with small holes, the principal object of which is to prevent the fracture of the bottles by isolating them from the bottom of the vessel, which is immediately exposed to the heat. On the movable bottom are soldered the cylinders *c c*, three or four centimetres in height, and above which, at the distance of eight centimetres, is a sheet of tin-plate, *p p*, pierced with ten holes, corresponding to the cylinders, and connected with the movable bottom by the supports, *s s*. These cylinders, and the sheet of tin-plate, are destined to isolate the bottles, *F F*, one from the other in the bath, and to keep them some time suspended over it, when the water is boiling, before complete immersion. The water-bath may be replaced by a steam-bath; the bottles will then be supported by a grating above the surface of the water. The solution of the alloy in the nitric acid takes place rapidly, and as it gives rise to an abundant evolution of nitrous vapour, it must be made under a flue having a good draught.

*Flue.*—This is represented at fig. 111. *C C* is a flue resting on a table or support, *T T*, about 90 centimeters high. The anterior side in the figure is removed to show the water-bath *B*, and the furnace *F*. The opening *O* of the flue is closed by the wooden door, *p*, movable on two excentric pivots, which keep it up during the solution, and allow it to fall so that the flask may be placed upon it. The nitrous vapour is removed from the bottles with the blower (fig. 94). The hood, *H*, prevents the diffusion of the nitrous vapour in the laboratory.

Fig. 111.



*Agitator.*—Figure 112 gives a sufficiently exact idea of this apparatus, and dispenses with a long description. It has ten cylindrical compartments, numbered from 1 to 10. The bottles, after solution of the alloy, are placed in it in the order of their numbers. The agitator is then placed by the side of the pipette, by which is measured the normal solution of salt, and into each flask is poured a pipetteful of the solution. The bottles are fitted with their stoppers, previously moistened with distilled water (fig. 113); they are then fixed in order with wooden wedges (fig. 114).

Fig. 112.

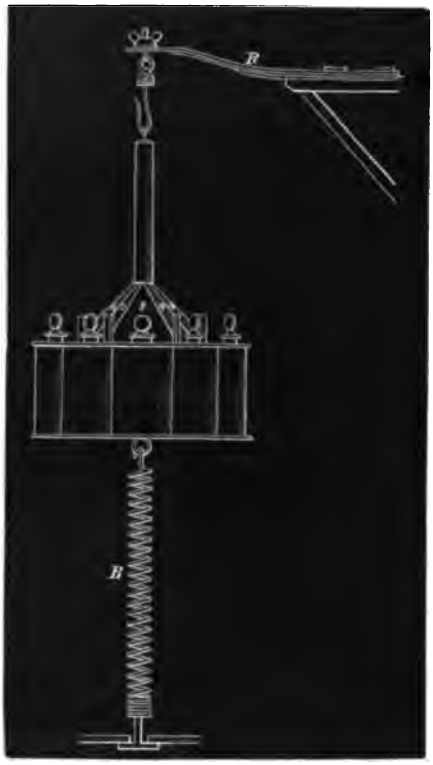


Fig. 113.

Fig. 114.

The agitator is suspended to a spring, *R*, and a rapid alternating movement given to it with both hands, by which the solution is agitated, and in less than a minute rendered as clear as water. This movement is assisted by a spiral spring, *B*, fixed to the agitator and its stand. The agitation finished, the wedges are removed, and placed in the vacant spaces between the compartments. The agitator is taken from the spring, and the bottles placed in order on a table prepared to receive them.

*Table.*—This table (fig. 115) has a double bottom; the upper is pierced with ten holes, a little larger than the diameter of the bottles, and of such a distance from the ower portion, or false bottom, that the flasks do not rise above its edge, or at least but little. This disposition is to

Fig. 115.

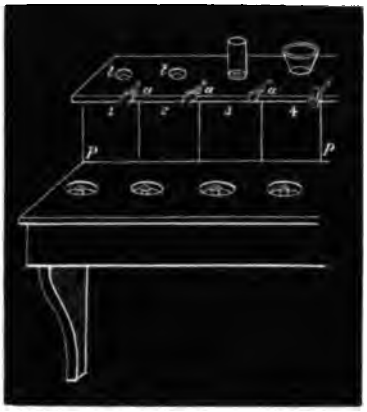
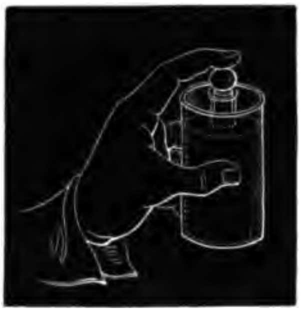
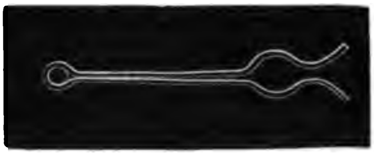


Fig. 116.



protect the chloride of silver from the light, for it decomposes in contact with water, and a little hydrochloric acid is produced, which requires for its precipitation a certain quantity of nitrate of silver, and so lowers the standard of the alloy. This cause of error is however not very great, at least when the light does not fall directly on the chloride; but it is easy to avoid, and should not be neglected. The disposition already pointed out does not at all complicate the process, and is moreover useful, as it prevents the fracture or upsetting of the bottles. When but one bottle is operated on, it is placed for agitation in a japanned tin-plate cylinder, which is held as shown at fig. 116. On placing the bottles in their respective places on the table, a brisk circular movement is given to them, so as to remove any chloride of silver adhering to the sides; their stoppers are removed and suspended by spring pincers, *a a*. These are formed of sheet-iron wire (see fig. 117). A thousandth of the decime solution is then poured into each bottle, and before this has been completed there will have formed in the first bottles where there is any precipitate, a well-marked

Fig. 117.

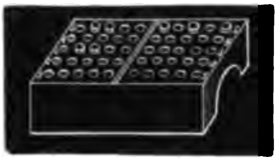


nebular layer about a centimetre in thickness. At the back of the table is a black board, *P P*, divided into compartments numbered from 1 to 10, on each of which is marked with chalk the number of thousandths of decime liquid added to the contents of the corresponding bottle. The thousandths of salt announcing augmentation of standard are preceded by the sign +, those of nitrate of silver by the sign −.

Lastly, the black board carries a small shelf pierced with holes, *t t*, and these receive the funnels or drain the bottles; on this shelf also are fastened the pincers for sustaining the stoppers.

*Cleansing the Bottles.*—The assays terminated the liquid from each flask is poured into a large vessel in which there is always a slight excess of common salt, and when it is full the clear supernatant fluid is removed by means of a syphon. Immediately will be given the means of reducing the chloride of silver so collected to the metallic state. The bottles, to the number of ten, are first rinsed with the same water passed from one to the other, then a second, and then a third time with fresh water. They are then placed to drain on the board just mentioned, and the stoppers are placed in a stand by series of tens (see figs. 118 and 107). It is important to remark, that when a glass has been rinsed with distilled water, care must be taken not to rub it with the fingers, for water poured in such a vessel would always be clouded on the addition of nitrate of silver. This effect is due to the chlorides contained in the perspiration, and is of course more to be guarded against in summer.

FIG. 118.



### *Reduction of Chloride of Silver, obtained in the Assay of Alloys by the Humid Method.*

Chloride of silver can be reduced without sensible loss, after having been well washed, by plunging into it scraps of iron or zinc, and adding dilute sulphuric acid in sufficient quantity to set up a slight disengagement of hydrogen gas.

The whole can be left to itself, and in the course of a few days the silver is completely reduced. This point can be easily determined by the colour and nature of the product, but better still by treating a small quantity by ammonia, which, if the chloride is perfectly reduced, will give no precipitate or cloudiness on treatment with an acid. The chlorine remains in solution in the water combined with zinc or iron. The residue must now be washed; the first washings are made with acidulated water, to dissolve oxide of iron which might have formed, and the following with ordinary water: after having completed the washing as much water as may be left is decanted, the mass dried, and a little powdered borax added. Nothing now remains but to fuse it. The powdered silver being voluminous, it is placed by separate portions into the crucible, in proportion as it sinks. The heat should be at first moderate, but towards the end of the operation should be sufficiently high to reduce the silver and slag to a state of complete liquidity. If it be found that not quite all the chloride was reduced by the iron or zinc, a little carbonate of potash or soda may be added to the powdered silver. The standard of silver thus obtained is from 999 to 1000 thousandths.

## *Preparation of Pure Silver.*

Take the silver prepared as above, dissolve it in nitric acid, and leave the solution some time in perfect rest in water, to deposit any gold it might contain. Decant the solution, and precipitate with common salt, well wash the precipitate, and reduce it, when the resulting silver will be pure.

M. Gay Lussac here gives a description of a process for the precipitation of chlorine from nitric acid for use in the mode of assay already described; but as that acid in a state of purity forms an ordinary article of commerce, and can be obtained at most operative chemists, the process will not be here reproduced.

### *Modifications required in the Assay of Silver Alloys containing Mercury.*

Whenever mercury is present in solution with silver, it is thrown down as insoluble chloride, and the assay rendered inaccurate. The presence of mercury in silver can be readily detected by the remarkable change which occurs in chloride of silver on exposure to light (viz. blackening) when free from mercury; but if the smallest quantity of the latter metal be present, no blackening will ensue. This source of error was removed by M. Levol in the following manner:—The sample being dissolved, as usual, in nitric acid, it was supersaturated with 25 cubic centimetres of caustic ammonia; then add the pipetteful of normal solution, and supersaturate the excess of ammonia with 20 cubic centimetres of acetic acid, and the operation continued in the usual way.

It may not be superfluous to state, that it is very easy to obtain an excellent result of an assay of silver containing mercury, made in the ordinary way, and in which the presence of the mercury is rendered manifest by the non-colouration of the precipitate under the influence of light. It suffices for this purpose to dissolve the precipitate in concentrated ammonia, and to supersaturate with acetic acid.

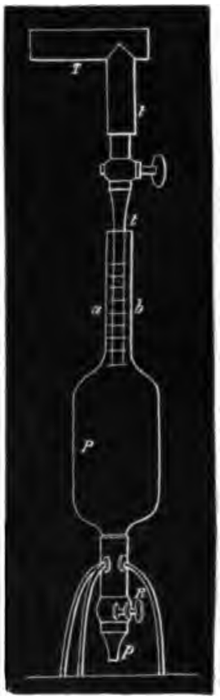
The ordinary acetic acid of commerce is employed, and the ammonia diluted with its volume of water, to avoid the too violent reaction. Both agents must be free from chlorides.

Some little time after the publication of this, M. Gay Lussac examined the above process himself, and very considerably simplified it. He says: 'After having confirmed by several experiments the accuracy of M. Levol's process, I thought it might be simplified by adding to the nitric solution of silver the ammonia and acetic acid at one and the same time, but in sufficient quantity to saturate the whole of the nitric acid, both that in combination with the silver and that in the free state. Ten grammes of acetate of ammonia were added, with a little water, to the silver dissolved in nitric acid, and the assay finished in the ordinary manner. The quantity indicated by synthesis was found very accurately, although 100 thousandths of mercury had been added.' Finally, M. Gay Lussac found that 10 grammes of acetate of soda, in crystals, also fully answered the purpose; and as that is a very cheap commercial salt, it is the best adapted for overcoming the difficulty in this class of assay, as regards the presence of mercury.

## APPENDIX.

In the foregone description of the method of assay by the humid method, it has been the object of the writer not to distract the attention by too numerous details. Here, however, will be given the processes to which personal experience has given the preference.

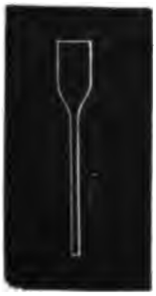
FIG. 119.



### *Apparatus for Weighing the Normal Solution of Salt.*

The apparatus about to be described enables the operator to weigh the normal solution of salt more rapidly than by means of the burette (fig. 82). It is a pipette, *P* (fig. 119), capable of furnishing in a continuous jet very nearly 100 grammes of solution, when filled up to the mark *a b*, at the ordinary temperature. As this weight changes its volume with the temperature, some marks are traced on the neck of the pipette, so as to regulate approximatively the volume to be taken. The pipette is terminated below by a three-footed stopcock, *R*, having a narrow outlet, *p* (about two millimetres). It is filled with solution by means of a small silver funnel (fig. 120), or better still by the suction tube, *T*, of the apparatus fig. 103, making an addition similar to that represented by fig. 118. The pipette is adjusted by absorbing the excess of liquid with a small roll of filtering or other absorbent paper, or by allowing its exit by the stopcock. The following is the method of proceeding:—

FIG. 120.



The pipette being approximatively adjusted to nearly 1 or 3 thousandths, it is placed in the balance described, fig. 87, with a constant equivalent weight, and the equilibrium effected by means of the rider. It is then placed over the bottle in which the assay was dissolved, the stopcock opened, and the liquid run out. The stopcock must be shut as soon as the jet stops. The pipette is again placed in the balance with a weight of 100 grains, and the equilibrium effected by aid of the rider. This process is certainly more rapid than weighing with the burette. The weighing can be made even more rapidly by suspending the burette from the beam of the balance.

### *Apparatus for Filling the Pipette with Normal Solution by Aspiration, and for convenient adjustment.*

This apparatus was the first employed, and has been in use for a considerable time. It is here described, because it appears extremely suitable for such persons as may be very little used to manipulation. It is sufficiently delineated in fig. 121. To fill the pipette, the jet or beak is plunged into a bottle containing the normal solution of salt, and the liquid is raised by the glass tube *T*, fixed to the socket *D* by means of a cork. The stopcock, *R*, is then closed whilst the tube is yet in the mouth, and the pipette is placed on its support in the following manner:—Take hold of its neck with the left hand, and place its beak or jet in the lower arm; then its neck in the upper arm, the blades of which are opened by means of the fingers. The pipette thus placed, so that its jet cannot be injured by the bottle, *F*, which is destined to receive the solution, it is adjusted by aid of the screw, *V*, whilst the 'handkerchief,' *M*, is applied to the jet; and as soon as it is adjusted, the handkerchief is removed with one hand, and the bottle placed under it with the other. The fluid is then allowed to run.

### *Another Apparatus for filling the Pipette with Normal Salt Solution.*

In this apparatus (fig. 122) the pipette is movable from below, above, to receive the tube *t*, through which the salt solution passes, and which fits the neck of the pipette like a funnel. To obtain this ascensional movement without lateral deviation, the jet of the pipette passes into a hole pierced in the cross piece *A B*, and the stopcock, fitted to its upper part, carries two wings, *R R*, working in slots cut in the supports, *M M*. The extent of movement is regulated by two corks, *b b*, cemented on to the lower part of the pipette.

FIG. 121.

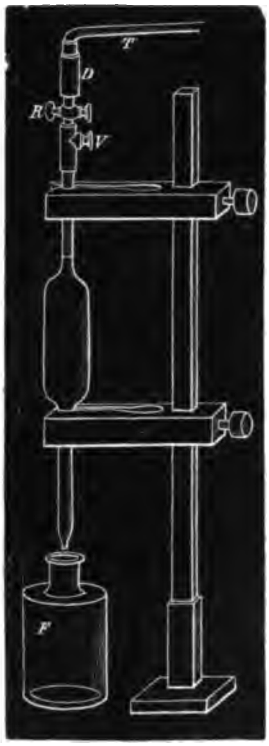
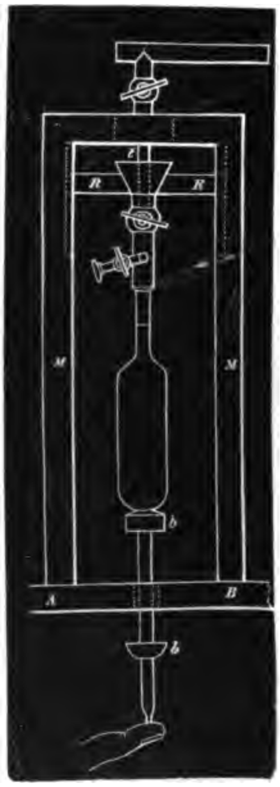


FIG. 122.



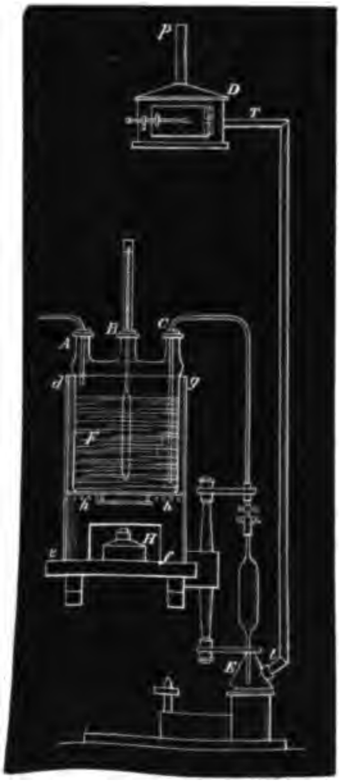
To fill it the forefinger of the left hand is placed against the lower orifice, and the whole raised until the cork, *b*, touches the cross piece. By this ascensional movement the tube, *t*, enters the neck of the pipette: immediately the stopcock of the reservoir must be opened. When the pipette is filled it is allowed to fall again, the stopcock shut, the finger removed, and the final adjustment made.

The reservoir containing the solution is, for the sake of convenience, movable.

### *Apparatus for Preserving the Normal Solution of Salt at a constant Temperature.*

The bulk of the normal solution of salt is too great to allow of its temperature being readily changed and reduced to any determinate degree. This, indeed, would be useless, for it suffices that the quantity of solution to be employed in the day should possess the desired temperature.

FIG. 123.



The solution, before entering the pipette from its reservoir, traverses an intermediate bottle, *F* (fig. 123), in which its temperature is suitably varied. The flask has three tubulures, *A*, *B*, *C*. To the tubulure *A* is adapted a tube with a stopcock; this carries the solution into the bottle. To the tubulure *B* is fixed a centigrade thermometer, which indicates the temperature of the solution; and through the tubulure *C* passes a syphon, which conveys the liquid to the pipette. The bottle is enveloped in a sheet-iron casing, *d e f g*, whose diameter is from three to four centimetres greater. The intermediate space is closed above by a border on the envelope, and by strips of paper cemented with glue. The bottle stands on a plate of sheet-iron of its own diameter, fixed to the casing by three supports; but is separated by a thick sheet of card-board, employed as a bad conductor of heat. Below this plate, at the distance of from 12 to 15 millimetres, is another of smaller diameter, the object of which is to deaden and spread the too powerful heat of a spirit-lamp, *H*, or, what is better, a gas flame, which is employed to raise the temperature of the salt solution. The heated air rises into the annular chimney, between the bottle and its casing, and escapes by the small circular openings, *h h*, &c. This apparatus only serves to heat the solution: it is very difficult to cool it.

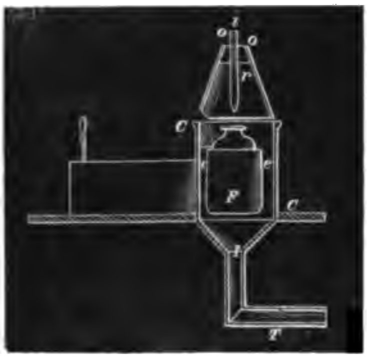
### *Means of Protection from the Nitrous Vapour disengaged from the Bottles during the Process of Assay by the Humid Method.*

After the solution of the silver in nitric acid, it has been recommended that the nitrous vapour be expelled from the flasks by the introduction of air by the blower, fig. 94. But the solution yet remains impregnated with nitrous vapour which continues to pass off; and it is only when it is completely cold that its disengagement is scarcely sensible. It therefore becomes necessary to find protection from this whilst the solution is yet very hot, and the vapour abundant. To the jet of the pipette, fig. 123, is adapted a funnel having a lateral tubulure, *t*, or simply an opening, by means of which it is placed in communication with a tube, *T*, *T*, of three or four centimetres in diameter, entering the case *D*, in which is a lamp, or a chauffer with live coals. The air necessary to support this combustion can only enter the box by passing through the funnel, and carries off the nitrous vapour displaced by the normal solution at the moment it is run into the bottle. From the case the nitrous vapour escapes with the air, by the tube *p*, either into a chimney or outside the laboratory. The funnel has a small portion cut off, so as to allow the free passage of the 'handkerchief' to the pipette.

This apparatus is very handy, and answers its purpose remarkably well; but, if the locality will allow, the following is preferable:—

The jet of the pipette also has a funnel (see fig. 124), but the draught is determined from below by means of the tube *T I*, which passes under the floor, and then enters the chimney or flue under which the alloys are dissolved. The cylinder, *e e*, in which the bottle, *F*, is placed, is enveloped in another cylinder, *C C*, two centimetres greater in diameter. It is through this intermediate space that the nitrous vapour is carried off. But, so that none may remain in the funnel, air also passes in by four openings, *o o*, pierced through the cork by which the funnel is fixed to the pipette. Lastly, in order to render the funnel easy of ascent and descent, a ferrule, *i r*, furnished exteriorly with a screw thread, is cemented to the beak of the pipette; and it is on this ferrule that the funnel turns. The interior cylinder is connected with the exterior by three small pieces of metal soldered to either cylinder, so as to leave the intermediate space as free as possible.*

Fig. 124.



### *Method of Taking the Assay from the Ingot.*

The ingots are so rarely perfectly homogeneous, even taking as a starting-point the standard 950 thousandths, that the differences remarked between the assays of samples made in different places should rather be attributed to the above cause than to the assay itself. It is important, therefore, to take a sample in a uniform manner, and from the same depth, on the upper surface of the ingot as on the lower. This condition is perfectly fulfilled by boring the ingots with a kind of drill, similar to that employed by the smith, and which is represented at fig. 125. The ingot, *L*, is placed in a copper tray, *C*; and in order to retain the borings, which might otherwise be thrown out, the drill, *f*, is surrounded by a casing, *m*, which does not impede its motion, and stands freely on the ingot. After a few turns of the drill, the first borings, which are not pure, are removed by means of a feather, and only those following are collected and reserved for assay. If it be desirable to try the lateral faces, it is necessary to employ a pressure screw, to keep the ingot in the position that may be deemed necessary.

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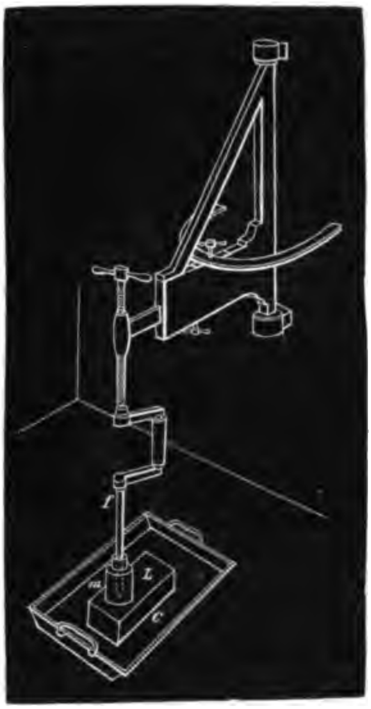
* A case built against the laboratory wall, having movable glazed windows in front, and connected with a flue, is the most simple mode of promoting the escape of noxious vapours.

FIG. 125.



The following is a slight modification of the process as already described, and the necessary apparatus are to be found in every laboratory. In this class of assay very simple apparatus are employed (similar to those used in alkalimetry) to determine the weight of the standard solution of salt added: and the results so obtained always correspond.

The apparatus employed are as follows:—A small flask for the solution of the silver to be assayed, a stoppered bottle (containing from three to four fluid ounces), in which the decomposition of the silver solution is made, and two small alkalimeters, known by the name of Schuster's. The alkalimeter is a light glass bottle, with two openings, one of which is drawn out, and extends over the side of the flask, parallel to its bottom; this aperture is for the purpose of allowing the fluid contained in the bottle to pass out in single drops, which it does most effectually. The other aperture just mentioned is furnished with a small stopper, and is used for the introduction of the fluid. An accurate balance and weights, with a few stirring rods, complete the set.

The standard solution of salt is made as follows:—It is absolutely necessary to employ pure salt. It is better to manufacture this, which may be accomplished by accurately neutralising pure hydrochloric acid with bicarbonate of soda, evaporating the solution to dryness, and fusing the dry residue, taking care to place it, whilst warm, in a well-stopped bottle, to preserve it perfectly free from moisture.

Distilled water must also be employed, of which 94·573 parts must have added to them 5·427 parts of salt, as above prepared. The solution so formed must be kept in glass stoppered bottles, and exposed as little as possible to the air during manipulation, otherwise it will become sensibly stronger by evaporation, and cause fallacious results; 100 grains, by weight, of this solution, precipitate exactly 10 grains of silver.

Another solution is also employed, the use of which will be pointed out hereafter. This solution is made by dissolving 10 grains of pure silver in a small quantity of nitric acid, and adding pure water (distilled) to the solution, until its weight amounts to 10,000 grains. This is the *verifying* solution.

This solution must be preserved with the same precautions as to exposure to air, &c., as the last; in addition to which it must be kept in a dark place.

The assay is thus made: 10 grains of the alloy are dissolved in nitric acid; when the solution is effected, water (distilled) must be added, and the whole poured into the assay bottle before mentioned. The flask in which the solution was made must be carefully washed out, and the rinsings added to that already in the assay bottle. A quantity (any amount) of the solution of salt must be placed in one of the alkalimeters, which, with it, must be carefully weighed, and the weight noted. The standard solution is now to be added, drop by drop, to the solution of the alloy in the bottle, replacing the stopper (taking care to hold it in the hand whilst dropping in the solution of salt) after each addition, and shaking the bottle well, to clarify its contents; repeating the above routine until the last drop occasions no turbidity in the liquid.

The weight of the alkalimeter and contents must now be again taken, and the amount of grains of salt solution employed noted.

There is most likely now in the bottle a little excess of salt, the amount of which must be estimated by the verifying solution just mentioned, in the following manner. Place in the other alkalimeter a certain amount (any quantity) of the verifying solution, and ascertain its weight with that of the alkalimeter, taking care to note it. Now add the solution, drop by drop, to the assay in the bottle, observing all the precautions as to agitation, &c., already pointed out, until the last drop causes no turbidity; then weigh the alkalimeter, and note the loss of weight, and from the amounts of solution used calculate the standard of the silver alloy in thousandths. This will be rendered perfectly clear by an example: 10 grains of alloy require for complete precipitation 60·7 grains of the salt solution; and as 10 grains of the solution are equal to 1 grain of silver, 60·7 is equal to 60·7 of silver; but a slight excess of solution has been added, which has increased the amount of silver above its true quantity; therefore 52 grains of the verifying solution were added; and as each 100 grains of such solution contains ·1 of a grain of silver, the 52 grains will contain ·052 of silver, which, deducted from 6·07 = 6·018 of silver, which gives 601·8 thousandths as the true standard of the alloy operated upon.

Mr. Sutton * has described the following modes of estimating silver:—

*Estimation of Silver, in Ores and Alloys, by Iodide of Starch. Methods of* PISANI *and* F. FIELD *(very accurate in the absence of mercury, protoxides, and salts of tin, iron, and manganese, antimony, arsenious acid, and chloride of gold).*

If a solution of the blue iodide of starch be added to a neutral solution of nitrate of silver, while any of the latter is in excess the blue colour disappears, the iodine entering into combination with the silver; as soon as all the silver is thus saturated the blue colour remains permanent and marks the end of the process; the reaction is very delicate, and the process accurate in the absence of the metals and salts mentioned above. It is more especially applicable to the analysis of ores and alloys of silver containing lead and copper.

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* *Handbook of Volumetric Analysis*, p. 188. London: Churchill.

The solution of iodide of starch devised by Pisani, is made by rubbing together in a mortar 2 gm. iodine with 15 gm. of starch and about 6 or 8 drops of water, putting the moist mixture into a stoppered flask and digesting in a water-bath for about an hour or until it has assumed a dark bluish-grey colour; water is then added till all is dissolved. The strength of the solution is then ascertained by titrating it with 10 c. c. of a solution of silver containing 1 gm. in the litre, to which a portion of pure precipitated carbonate of lime is added; the addition of this latter removes all excess of acid, and at the same time enables the operator to distinguish the end of the reaction more accurately. The starch solution should be of such a strength that about 50 c. c. is required for 10 c. c. of the silver solution (=0·01 gm. silver).

F. Field,* who discovered the principle of this method simultaneously with Pisani, uses a solution of iodine in iodide of potassium with starch liquor. Those who desire to make use of this plan can use deci- and centi-normal solutions of iodine, the results being the same in either case.

In the analysis of silver containing copper the solution must be considerably diluted in order to weaken the colour of the copper, a small measured portion is then taken, carbonate of lime added, and iodide of starch till the colour is permanent. It is best to operate with about from 60 to 100 c. c., containing not more than 0·02 gm. silver; when the quantity is much greater than this it is preferable to precipitate the greater portion with $\frac{N}{10}$ chloride of sodium, and to complete with iodide of starch after filtering off the chloride. When lead is present with silver in the nitric acid solution, add sulphuric acid and filter off the sulphate of lead, then add carbonate of lime to neutralise excess of acid, filter again, if necessary, then add fresh carbonate of lime and titrate as above.

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* *Chem. News*, vol. ii. p. 17.

### *Assay of Commercial Silver (Plate, Bullion, Coin, &c.).*
### GAY-LUSSAC'S *Method modified by* J. G. MULDER.

For more than thirty years Gay-Lussac's method of estimating silver in its alloys has been practised intact, at all the European mints, under the name of the 'humid method,' in place of the whole system of cupellation; during that time it has been regarded as one of the most exact methods of quantitative analysis; the researches of Mulder, however, into the innermost details of the process have shown that it is capable of even greater accuracy than has hitherto been gained by it. For the particulars of Mulder's investigations I cannot do better than refer the reader to the excellent translation of his memoir, published in the 'Chemical News,' by Dr. Adriani.

The principle of the process is the affinity which chlorine has for silver in preference to all other substances, and resulting in the formation of chloride of silver, a compound insoluble in dilute acids, and which readily separates itself from the liquid in which it is suspended.

The plan originally devised by the illustrious inventor of this process for assaying silver, and which is still followed, is to consider the weight of alloy taken for examination to consist of 1000 parts, and the question is to find how many of these parts are pure silver. This empirical system was arranged for the convenience of commerce, and being now thoroughly established it is the best plan of procedure; if, therefore, standard solution of salt be made of such strength that 100 c.c. will exactly precipitate 1 gramme of silver, it is manifest that each $\frac{1}{10}$ c. c. will precipitate 1 milligramme or $\frac{1}{1000}$th part of the gramme taken, and consequently in the analysis of 1 gramme of any alloy containing silver, the number of $\frac{1}{10}$ c. c. required to precipitate all the silver out of it would be the number of thousandths of pure silver contained in the specimen.

In practice, however, it would not do to follow this plan precisely, inasmuch as neither the measurement of the standard solution nor the ending of the process would be gained in the most exact manner, consequently a decimal solution of salt, one-tenth the strength of the standard solution, is prepared, so that 1000 c. c. will exactly precipitate 1 gramme of silver, and, therefore, 1 c. c. one milligramme.

The silver alloy to be examined (the composition of which must be approximately known) is weighed so that about 1 gramme of pure silver is present; it is then dissolved in pure nitric acid by the aid of a gentle heat, and 100 c. c. of standard solution of salt added from a pipette in order to precipitate exactly 1 gm. of silver, the bottle containing the mixture is then well shaken until the chloride of silver has curdled, leaving the liquid clear.

The question is now—Which is in excess, salt or silver? A drop of decimal salt solution is added, and if a precipitate is produced, 1 c. c. is delivered in, and after clearing, another, and so on, as long as a precipitate is produced; if, on the other hand, the one drop of salt produced no precipitate, showing that the pure silver present was less than 1 gm., a decimal solution of silver is used, prepared by dissolving 1 gm. pure silver in pure nitric acid and diluting to 1 litre; this solution is added after the same manner as the salt solution just described, until no further precipitate occurs; in either case the quantity of decimal solution used is noted, and the results calculated in thousandths for 1 gm. of the alloy.

The process thus shortly described is that originally devised by Gay-Lussac, and it was taken for granted that when equivalent chemical proportions of silver and chloride of sodium were brought thus in contact that every trace of the metal was precipitated from the solution, leaving nitrate of soda and free nitric acid only in solution. The researches of Mulder, however, go to prove that this is not strictly the case, but that when the most exact chemical proportions of silver and salt are made to react on each other, and the chloride has subsided, a few drops more of either salt or silver solution will produce a further precipitate, indicating the presence of both nitrate of silver and chloride of sodium in a state of equilibrium, which is upset on the addition of either salt or silver. Mulder decides, and no doubt rightly, that this peculiarity is owing to the presence of nitrate of soda, and varies somewhat with the temperature and state of dilution of the liquid.

It therefore follows that when a silver solution is carefully precipitated, first by concentrated and then by dilute salt solution, until no further precipitate appears, the clear liquid will at this point give a precipitate with dilute silver solution, and if this be added till no further cloudiness is produced, it will again be precipitable by dilute salt solution.

For example: suppose that in a given silver analysis the decimal salt solution has been added so long as a precipitate is produced, and that 1 c. c. (=20 drops of Mulder's dropping apparatus) of decimal silver is in turn required to precipitate the apparent excess, it would be found that when this had been done, 1 c. c. more of salt solution would be wanted to reach the point at which no further cloudiness is produced by it, and so the changes might be rung time after time; if, however, instead of the last 1 c. c. (=20 drops) of salt, half the quantity be added, that is to say 10 drops (=½ c. c.); Mulder's so-called neutral point is reached, namely, that in which, if the liquid be divided in half, both salt and silver will produce the same amount of precipitate. At this stage the solution contains chloride of silver dissolved in nitrate of soda, and the addition of either salt or silver expels it from solution.

A silver analysis may therefore be concluded in three ways—

1. By adding decimal salt solution until it just ceases to produce a cloudiness.

2. By adding a slight excess of salt, and then decimal silver till no more precipitate occurs.

3. By finding the neutral point.

According to Mulder, the latter is the only correct method, and preserves its accuracy at all temperatures up to 56° C.

(=133° Fahr.), while the difference between 1 and 3 amounts to ½ a milligramme, and that between 1 and 2 to 1 milligramme on 1 gramme of silver at 16° C. (=62° Fahr.), and is seriously increased by variation of temperature.

It will readily be seen that much more trouble and care is required by Mulder's method than by that of Gay-Lussac, but as a compensation, much greater accuracy is obtained.

On the whole, it appears to me preferable to weigh the alloy so that slightly more than 1 gm. of silver is present, and to choose the ending No. 1, adding drop by drop the decimal salt solution until just a trace of precipitate is seen, and which, after some practice, is known by the operator to be final. It will be found that the quantity of salt solution used will slightly exceed that required by chemical computation—say 100·1 c.c. are found equal to 1 gm. silver, the operator has only to calculate that quantity of the salt solution in question for every 1 gm. silver he assays in the form of alloy, and the error produced by the solubility of chloride of silver in nitrate of soda is removed.

If the decimal solution has been cautiously added, and the temperature not higher than 62° Fahr., this method of conclusion is as reliable as No. 3, and free from the possible errors of experiment, for it requires a great expenditure of time and patience to reverse an assay two or three times, and each time cautiously adding the solutions, drop by drop, then shaking and waiting for the liquid to clear, beside the risk of discolouring the chloride of silver, which would at once vitiate the results.

The decimal silver solution, according to this arrangement, would seldom be required; if the salt has been incautiously added, or the quantity of alloy too little to contain 1 gm. pure silver, then it is best to add once for all 2, 3, or 5 c.c. according to circumstances, and finish with decimal salt as No. 1, deducting the silver added.

### *The Standard Solutions and Necessary Apparatus.*

*a. Standard Solution of Salt.*—Pure chloride of sodium is prepared by treating a concentrated solution of the whitest table salt first with a solution of caustic baryta to remove sulphuric acid and magnesia, then with a slight excess of carbonate of soda to remove baryta and lime, warming and allowing the precipitates to subside; then evaporating to a small bulk, that crystals may form; these are separated by a filter, and slightly washed with cold distilled water; dried, removed from the filter, and heated to a dull redness, and when cold preserved in a well-closed bottle for use. The mother liquor is thrown away or used for other purposes. Of the salt so prepared, or of chemically pure rock-salt (steinsalz, a substance to be obtained freely in Germany) 5·4145 gm. is to be weighed and dissolved in 1 litre of distilled water at 62° Fahr. 100 c. c. of this solution will precipitate exactly 1 gm. silver; it is preserved in a well-stoppered bottle, and shaken before use.

*Decimal Solution of Salt.*—100 c. c. of the above solution is diluted to exactly 1 litre with distilled water at 62° Fahr. 1 c. c. will precipitate 0·001 gm. silver.

*b. Decimal Solution of Silver.*—Pure metallic silver is best prepared by galvanic action from pure chloride; and as clean and secure a method as any, is to wrap a lump of clean zinc, into which a silver wire is melted, with a piece of wetted bladder or calico, so as to keep any particles of impurity contained in the zinc from the silver. The chloride is placed at the bottom of a porcelain dish, covered with dilute sulphuric acid, and the zinc laid in the middle; the silver wire is bent over so as to be immersed in the chloride; as soon as the acid begins to act upon the zinc, the reduction commences in the chloride and grows gradually all over the mass; the resulting finely-divided silver is well washed, first with dilute acid, then with hot water, till all acid and soluble zinc are removed.

The moist metal is then mixed with a little carbonate of soda, saltpetre, and borax, say about an eighth part of each, and dried perfectly.

The metallic silver obtained as above is never free altogether from organic matter and undecomposed chloride, and, therefore, it must invariably be melted. Mulder recommends that the melting should be done in a porcelain crucible, immersed in sand contained in a common earthen crucible; borax is sprinkled over the surface of the sand so that it may be somewhat vitrified, that in pouring out the silver when melted no particles of dirt or sand may fall into it. If the quantity of metal is small it may be melted in a porcelain crucible over a gas blowpipe.

The molten metal obtained in either case can be poured into cold water and so granulated, or upon a slab of pipe-clay, into which a glass plate has been pressed when soft so as to form a shallow mould. The metal is then washed well with boiling water to remove accidental surface impurities, and rolled into thin strips by a goldsmith's mill, in order that it may be readily cut for weighing; the granulated metal is, of course, ready for use at once without any rolling.

1 gm. of this silver is dissolved in pure dilute nitric acid and diluted to 1 litre—each c.c. contains 0·001 gm. silver—it should be kept from the light.

*Dropping Apparatus for concluding the Assay.*—Mulder constructs a special affair for this purpose, consisting of a pear-shaped vessel fixed in a stand, with special arrangements for preventing any continued flow of liquid; the delivery tube has an opening of such size that 20 drops measure exactly 1 c.c.—the vessel itself is not graduated. As this arrangement is of more service to assay than to general laboratories, it need not be further described here. A small burette divided in $\frac{1}{10}$ c.c. with a convenient dropping tube will answer every purpose, and possesses the further advantage of recording the actual volume of fluid delivered.

The 100 c.c. pipette, for delivering the concentrated salt solution, must be accurately graduated, and should deliver exactly 100 gm. of distilled water at 62° Fahr.

The test bottles, holding about 200 c. c. should have their stoppers well ground and brought to a point, and should be fitted into japanned tin tubes reaching as high as the neck, so as to preserve the precipitated chloride from the action of light, and, when shaken, a piece of black cloth should be covered over the stopper.

*c. Titration of the Standard Salt Solution.*—It is not possible to rely absolutely upon a standard solution of salt, containing 5·4145 gm. per litre, although this is chemically correct in its strength. The real working power must be found by experiment. From 1·002 to 1·004 gm. of absolutely pure silver is weighed on the assay balance, put into a test bottle with about 5 c. c. of pure nitric acid about 1·2 spec. grav., and gently heated in the water or sand bath till it is all dissolved. The nitrous vapours are then blown from the bottle, and it is set aside to cool down to about 62° Fahr.

The 100 c. c. pipette, which should be securely fixed in a support, is then carefully filled with the salt solution, and delivered into the test bottle contained in its case, the moistened stopper inserted, covered over with the black velvet or cloth, and shaken continuously till the chloride has clotted and the liquid becomes clear; the stopper is then slightly lifted and its point touched against the neck of the bottle to remove excess of liquid, again inserted, and any particles of chloride washed down from the top of the bottle by carefully shaking the clear liquid over them. The bottle is then brought under the decimal salt burette, and ½ c. c. added, the mixture shaken, cleared, another ½ c. c. put in, and the bottle lifted partly out of its case to see if the precipitate is considerable; lastly, 2 or 3 drops only of the solution are added at a time until no further opacity is produced by the final drop. Suppose, for instance, that in titrating the salt solution it is found that 1·003 gm. silver require 100 c. c. concentrated, and 4 c. c. decimal solution, altogether equal to 100·4 c. c. concentrated, then—

$$1{\cdot}003 \text{ silver} : 100{\cdot}4 \text{ salt} :: 1{\cdot}000 : x = 100{\cdot}0999.$$

The result is within $\frac{1}{10,000}$ of 100·1, which is near enough for the purpose and may be more conveniently used. The operator, therefore, knows that 100·1 c. c. of the concentrated salt solution at 62° Fahr. will exactly precipitate 1 gm. silver, and calculates accordingly in his examination of alloys.

In the assay of coin and plate of the English standard, namely, 11·1 silver and 0·9 copper, the weight corresponding to 1 gm. silver is 1·081 gm., therefore, in examining this alloy 1·085 gm. may be weighed.

When the quantity of silver is not approximately known, a preliminary analysis is necessary, which is best made by dissolving $\frac{1}{2}$ or 1 gm. of the alloy in nitric acid, and precipitating very carefully with the concentrated salt solution from a $\frac{1}{10}$ c. c. burette. Suppose that in this manner 1 gm. of alloy required 45 c. c. salt solution,

100·1 salt : 1·000 silver 45 : $x$=0·4495, and again
0·4495 : 1 :: 1·003 : $x$=2·231.

2·231 gm. of this particular alloy are therefore taken for the assay.

Where alloys of silver contain sulphur or gold, with small quantities of tin, lead, or antimony, they are first treated with a small quantity of nitric acid so long as red vapours are disengaged, then boiled with concentrated sulphuric acid till the gold has become compact, set aside to cool, diluted with water, and titrated as above.

## BLOWPIPE REACTIONS OF SILVER.

### ORES OF SILVER.

**Sulphide of Silver.**—*Alone*, on charcoal, fuses and swells considerably, forming large bubbles; but after a continued blast, it forms a grain. It gives off an odour of sulphurous acid, and finally furnishes a grain of silver, surrounded by slag. Fused with borax and microcosmic salt, the slag gives traces of copper.

**Red Silver.**—*Alone*, on charcoal, decrepitates little, fuses, burns and smokes, like antimony, but gives no arsenical odour. The production of vapour lasts but for a few minutes.

In the *open tube*, it gives much vapour, and a smell of sulphurous acid, which is very strong at the commencement. The deposit on the sides of the tube is sometimes crystalline; it is oxide of antimony. The bead which remains after a long exposure to the exterior flame is a button of pure silver.

**Antimonial Silver, and Argentiferous Antimony.**—*Alone*, on charcoal, fuses readily, forming a metallic bead, which is not malleable, giving off a vapour like that of pure antimony, but less abundant. The bead becomes, after the disengagement of a certain quantity of antimony, dull white, and very crystalline, entering into ignition at the instant of congelation. When it has lost still more antimony, its surface becomes smooth, like glass; and the heat which it then disengages is more intense than at any other time. Lastly, after a long-continued blast, nothing but pure silver remains.

In the tube, much oxide of antimony is given off, and the bead which remains is surrounded by a bead of deep yellow glass.

**Electrum** gives by fusion a grain of gold, which varies in whiteness, and which gives with borax and microcosmic salt the same reactions as pure silver.

**Amalgam**, in the matrass, swells up, and gives much mercury, leaving silver, which may be fused to a bead on charcoal.

**Chloride of Silver, Horn Silver.**—On charcoal becomes a bead, which, according to the purity of the salt, is grey, brownish, or black. In the reducing flame it is gradually converted into metallic silver. It gives with microcosmic salt, fused on the platinum wire, a blue flame, like the chloride of mercury.

**Oxide of Silver.**—*Alone*, is reduced instantaneously.

*With borax* a part is dissolved and a part reduced. In the oxidising flame the glass becomes, on cooling, milk-white, taking the colours of the opal, according to the quantity of the silver dissolved. In the reducing flame it becomes greyish, owing to the dissemination of particles of metallic silver.

*With microcosmic salt* the oxide and the metal give in the oxidising flame a yellowish opaline glass; seen by refraction, in the day it appears yellow; seen in the same manner by the light of the lamp it appears reddish.

This is the most appropriate place to introduce the valuable and highly interesting researches on the application of the blowpipe to the assay of silver, by David

Forbes, F.R.S., as given in the *Chemical News*, Nos. 380, 384, 392, 396, 398, and 412.

The blowpipe assay of silver ores was first described in 1827 by Harkort,* and subsequently considerably improved by Plattner. This assay process is in all cases based upon the reduction to a metallic state of all the silver contained in the compound in question along with more or less metallic lead, which latter metal, when not already present in sufficient quantity in the substance itself under examination, is added in the state of granulated lead to the assay previous to its reduction. The globule of silver-lead thus obtained, if soft and free from such elements as would interfere with its treatment upon the cupel, may then be at once cupelled before the blowpipe until the pure silver alone remains upon the bone ash surface of the cupel; but if not, it is previously submitted to a scorifying or oxidating treatment upon charcoal until all such substances are either slagged off or volatilised, and the resulting silver-lead globule cupelled as before.

As, therefore, the final operation in all silver assays is invariably that of cupelling the silver-lead alloy obtained from the previous reduction of the substance, effected by methods differing according to the nature of the argentiferous ore or compound under examination, it is here considered advisable to introduce the description of the silver assay by an explanation of this process.

In the ordinary process of cupellation in the muffle, bone-ash or other cupels are employed of a size large enough to absorb the whole of the litharge produced from the oxidation of the lead in the assay.

This, however, should not be the case when using the blowpipe; for as the heating powers of that instrument are limited, it is found in practice much better to accomplish this result by two distinct operations—the first being a concentration of the silver-lead in which the greater part of the lead is converted by oxidation into litharge remaining upon, but not, or only very slightly, absorbed by, the bone-ash cupel; and the second in cupelling the small concentrated metallic bead so obtained upon a fresh cupel until the remaining lead is totally absorbed by the cupel and the silver left behind in a pure state. By this means a much larger weight of the silver-lead alloy can be submitted to assay, and, for reasons hereafter to be explained, much more exact results are obtained than would be the case when the cupellation is conducted at one operation in the ordinary manner.

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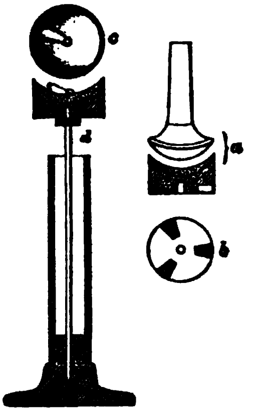
* *Die Probirkunst mit dem Löthrohre.* Freiberg, 1827, I. Heft (all published).

The apparatus used by the author for these operations are shown to a scale of one-half their real size in the woodcuts fig. 126 (*a* to *d*).

FIG. 126.



In fig. 126 *a* represents in section a small cylindrical mould of iron, seven-tenths of an inch in diameter, and about four-tenths high, in which is turned a cup-shaped nearly hemispherical depression two-tenths of an inch deep in centre, the inner surface of which is left rough, or marked with minute ridges and furrows for the purpose of enabling it to retain more firmly the bone-ash lining which is stamped into it by means of the polished bolt, also shown in the figure. This mould rests upon the stand *d*, having for this purpose a small central socket in its base, into which the central pivot of the stand enters. This socket is seen in the ground plan *b* of the base of the mould, which shows likewise three small grooves or slots made to enable a steady hold to be taken of it, when hot, by the forceps. The stand itself is composed of a small turned ivory or wood base, fixed into a short piece of strong glass tubing, which, from its non-conducting powers, serves as an excellent handle. In the centre of the base a slight iron rod rising above the level of the glass outer tube serves as a support for the cupel mould, into the socket in the base of which it enters.

Bone ash is best prepared by burning bones which previously had been boiled several times, so as to extract all animal matter. The best bone ash is made from the core-bone of the horns of cattle well boiled out and burned. The ash from this is more uniform than from the other bones, which have in general a very compact enamel-like exterior surface, whilst the interior is of a much softer nature.

*Concentration of the Silver-lead.*—A cupel is prepared by filling the above described cupel mould with bone-ash powder not finer than will pass through a sieve containing from forty to fifty holes in the linear inch, and should be well dried and kept in an airtight bottle, and the whole pressed down with the bolt, using a few taps of the hammer. It is then heated strongly in the oxidating blowpipe flame, in order to drive off any hygroscopic moisture. The bone-ash surface of the cupel, after heating, should be smooth, and present no cracks; if the reverse, these may be removed by using the bolt again and reheating.* The silver-lead, beaten on the anvil into the form of a cube, is placed gently upon the surface of the bone ash, and, directing a pretty strong oxidating flame on to its surface, it is fused, and quickly attains a bright metallic appearance, and commences to oxidise with a rapid rotatory movement. (Occasionally, when the assay is large, and much copper or nickel present, the globule may, under this operation, cover itself with a crust of oxide of lead or solidify; in such cases direct the blue point of a strong flame steadily on to one spot on the surface of the lead globule until it commences oxidating and rotating. In some cases where much nickel is present, an infusible scale, impeding or even preventing this action, may form, but will disappear on adding more lead—say from three to six grains, according to the thickness of this scale or crust.) When this occurs, the cupel is slightly inclined from the lamp, and a fine blue point obtained by placing the blowpipe nozzle deeper into the flame, and the lamp is directed at about an angle of 30° on to the globule—not, however, so near as to touch it with the blue point, but only with the outer flame, so moderating it as to keep the assay at a gentle red heat, and not allowing the rotation to become too violent.

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* These precautions are very important, as the slightest trace of moisture in the substance of the bone ash would inevitably cause a spirting of the metal during the operation.

This oxidating fusion should be carried on at the lowest temperature sufficient to keep up the rotatory movement, and to prevent a crust of litharge accumulating upon the surface of the globule, but still sufficiently high to hinder the metallic globule from solidifying. Should this, however, happen, a stronger flame must be employed for a moment until the metal is again in rotation ; such interruptions should, however, be avoided. The proper temperature can only be learned by practice; a too high temperature is still more injurious, causing the lead to volatilise, and, if rich in silver, carry some of that metal mechanically along with it. The litharge also, instead of remaining on the cupel, would be absorbed by the bone ash, and as the surface of the metallic globule is covered by a too thin coating of fused litharge, some silver may be absorbed along with the litharge. In this operation, in order to avoid loss of silver, the fused globule should be always kept in contact with the melted litharge.

By the above treatment, the air has free access to the assay, and the oxidation of the lead and associated foreign metals goes on rapidly. The surface of the melted globule, when poor in silver, shows a brilliant play of iridescent colours, which does not take place when very rich in silver. The litharge is driven to the edge of the globule, heaping itself up and solidifying behind and around it. When the globule becomes so hemmed in by the litharge as to present too small a surface for oxidation, the cupel is moved so as to be more horizontal (having been previously kept on an inclined position), thus causing the lead globule to slide by its own weight on one side, and expose a fresh surface to the oxidising action. When the lead is pure, the litharge formed has a reddish-yellow colour, but, if copper is present, is nearly black.

In concentrating silver-lead, it must be remembered that an alloy of lead and silver, if in the proportion of about 86 per cent. silver along with 14 per cent. lead, when cooled slowly in the litharge behaves in a manner analogous to the spitting of pure silver, throwing out a whitish-grey pulverulent excrescence rich in silver. For this reason, therefore, the concentration above described should be stopped when the globule is supposed to contain about six parts silver along with one part in weight of lead. In case, however, this limit should have been exceeded, it is advisable at once to push the concentration still further until the silver globule contains but very little lead. In practice with poor ores it is usual to concentrate the lead until the globule is reduced to the size of a small mustard seed, or in rich ores to some two or three times that size. Upon arriving at this point, the cupel is withdrawn very gradually from the flame, so that the cooling shall take place as slowly as possible until the globule has solidified in its envelope of litharge. If cooled too quickly, the litharge, contracting suddenly, would throw out the globule, or even cause it to spirt; in such case it should be touched by the point of the blue flame so as to fuse it to a round globule, which is cooled slowly, as before described. The globule is now reserved for the next operation, for which purpose it is, when quite cold, extracted from the litharge surrounding it.

*Cupellation.*—The bone ash required for this process should be of the best quality and in the most impalpable powder, prepared by elutriating finely-ground bone ash, and drying the product before use.

The cupel, still hot from the last operation, is placed upon the anvil, and the crust of litharge, with its enclosed metallic bead, gently removed, leaving the hot coarse bone ash beneath it in the mould; upon this a small quantity of the elutriated bone ash is placed, so as to fill up the cavity, and the whole, whilst hot, stamped down by the bolt, previously slightly warmed, with a few taps of the hammer. The cupel thus formed is heated strongly in the oxidating flame, which should leave the surface perfectly smooth, and free from any fissures or scales; if such appear, the bolt must again be used, and the cupel re-heated. In this process it is very important that the cupel should possess as smooth a surface as possible, whilst at the same time the substance of the cupel beneath should not be too compact, so as thereby to permit the litharge to filter through and be readily absorbed, leaving the silver bead upon the smooth upper surface.

The bead of silver-lead obtained from the last operation is taken out of the litharge in which it is embedded, and, after removing any trace of adherent bone ash or litharge, is slightly flattened to prevent its rolling about upon the surface of the cupel.

It is now put into the cupel prepared as before described, placing it on the side furthest from the lamp and a little above the centre of the cupel, which is now inclined slightly towards the lamp, and is heated by the oxidating flame directed downwards upon it, this causing the globule, when fused and oxidating, to move of itself into the centre of the cupel. The cupel is now brought into a horizontal position, and the flame, directed on to it at an angle of about forty-five degrees, is made to play upon the bone-ash surface immediately surrounding the globule, without, however, touching it, so as to keep this part of the cupel at a red heat sufficiently strong to insure the globule being in constant oxidising fusion, at the same time to cause the perfect absorption of the litharge, so as to prevent any scales of litharge forming upon the surface of the cupel under the globule, which would impede the oxidation, as well as prevent the silver bead being easily detached at the conclusion of the operation. Should the heat at any time be too low and the globule solidify, it must be touched for an instant with the point of the flame and proceeded with as before. Should (in consequence of the bone ash not having been sufficiently heated to absorb the litharge perfectly) a little litharge adhere pertinaciously to the globule, or a particle of the bone-ash cupel attach itself, the cupel should be slightly inclined, so as to allow the globule to move by its own weight on to another and clean part of the cupel, leaving the litharge or bone ash behind it; but, if not sufficiently heavy to do so, a small piece of pure lead may be fused to it in order to increase its weight, and so allow of the same proceeding being adopted.

By slightly inclining the cupel stand, and moving it so as to present in turn all parts of the surface surrounding the globule to the action of the flame, the cupellation proceeds rapidly. If, however, the assay contains very little silver, it will be found necessary to move the globule from one spot to another on the cupel, in order to present a fresh surface for absorbing the litharge formed; this is done by simply inclining the cupel stand, remembering that the bone ash surrounding the globule must always be kept at a red heat, without ever touching the globule itself by the flame.

In assays rich in silver a play of iridescent colours appears some seconds before the 'brightening,' which disappears the moment the silver becomes pure; as soon as this is observed the cupel should be moved in a circular manner, so that the globule is nearly touched all round by the point of the blue flame, and this is continued until the surface of the melted silver is seen to be quite free from any litharge, upon which it is very gradually withdrawn from the flame so as to cool the assay by degrees very slowly, in order to prevent 'spitting.'

When the silver-lead is very poor, this play of colours is not apparent, and as soon as the rotatory movement of the globule ceases, the heat must be increased for an instant, in order to remove the last thin but pertinacious film or scale of litharge, and subsequently the assay is cooled gradually; when cold it should, whilst still upon the cupel, be examined by a lens, to see whether the bead possesses a pure silver colour, as, if not, it must be re-heated.

Frequently, when the 'brightening' takes place, the silver globule is found to spread out, and, after cooling, although of a white colour, is found to appear somewhat less spherical or more flattened in shape than a corresponding globule of pure silver would be. This arises from the presence of copper still remaining in the silver, and in such cases a small piece of pure lead (about from one-half to one and a half grain in weight, according to size of assay) should be fused on the cupel along with the silver, and the cupellation of the whole conducted as before on another part of the cupel, when the silver globule will be obtained pure, and nearly spherical in shape. Sometimes the silver globule in 'brightening' may still remain covered with a thin film of litharge, although otherwise pure; this arises from too little heat having been employed in the last stage of the operation, and consequently the bead should be re-heated in a strong oxidating flame until this litharge is absorbed, and the globule, after slow cooling, appears pure.

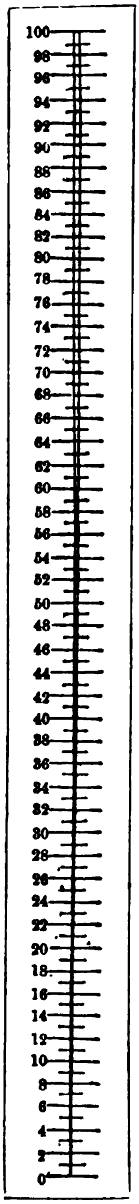
If the instructions here given be strictly attended to, it will be found after some practice, that very accurate results may be obtained in the blowpipe assay for silver, and that no difficulty will be found in detecting the presence and determining the amount of silver present, even when in as small a quantity as half an ounce to the ton. When substances containing very little silver or less than that amount are examined, several assays should be made, and the silver-lead obtained concentrated separately, after which the various globules should be united and cupelled together in one operation.

It is hardly necessary to remark, that the lead employed in assaying should be free from silver, or if not, its actual contents in silver should be determined, and subtracted from the amount found in the assay.

Assay lead containing less than one quarter of an ounce to the ton of lead can readily be obtained, or can be made by precipitating a solution of acetate of lead by metallic zinc, rejecting the first portion of lead thrown down. In all cases the lead should be fused and granulated finely—the granulated lead for use in these assays being previously passed through a sieve containing forty holes to the linear inch. It is also useful to have some lead in the form of wire, as being being very convenient for adding in small portions to assays when on the cupel.

*Determination of the Weight of the Silver Globule obtained on Cupellation.*—As the amount of lead which can, by the method before described, be conveniently cupelled before the blowpipe, is necessarily limited, the silver globule which remains upon the bone-ash surface of the cupel at the end of the operation is, when substances poor in silver have been examined, frequently so very minute that its weight could not be determined with correctness by the most delicate balances in general use.

Fig. 127.



The blowpipe balance employed by the author turns readily with one-thousandth of a grain, but could not be used for determining weights below that amount.

Globules of silver of far less weight than one-thousandth are distinctly visible to the naked eye—a circumstance which induced Harkort to invent a volumetrical scale based upon the measurement of the diameters of the globules, which scale in practice has been found of very great utility in the blowpipe assay of silver.

The scale for this purpose which is employed by the author is shown in full size in the annexed woodcut.

This figure represents a small strip of highly polished ivory about 6½ inches long, ⅜ inch broad, and ⅛ inch in thickness, on which are drawn, by an extremely fine point, two very fine and distinct lines emanating from the lower or zero point, and diverging upwards until, at the distance of exactly six English standard inches, they are precisely four-hundredth parts of an inch apart. This distance (six inches) is, as shown in woodcut, divided into 100 equal parts by cross lines numbered in accordance from zero upwards. It is now evident, if a small globule of silver be placed in the space between these two lines, using a magnifying glass to assist the eye in moving it up or down until the diameter of the globule is exactly contained within the lines themselves, that we have at once a means of estimating the diameter of the globule itself, and therefrom are enabled to calculate its weight.

As the silver globules which cool upon the surface of the bone-ash cupel are not true spheres, but are considerably flattened on the lower surface, where they touch and rest upon the cupel, it follows that the weight of globules corresponding in diameter to the extent of divergence at the different degrees of the scale cannot be calculated directly from their diameters as spheres, but require to have their actual weight experimentally determined in the same manner as employed by Plattner.

The table here appended has been calculated by the author, and in one column shows the diameter in English inches corresponding to each number or degree of the scale itself, and in the two next columns the respective weights of the flattened spheres which correspond to each degree or diameter; for convenience these weights are given in the different columns in decimals, both of English grains and of French grammes.

These weights are calculated from the following data, found as the average result of several very careful and closely approximating assays, which showed that globules of silver exactly corresponding to No. 95 on this scale, or 0·038 inch in diameter, possessed a weight of 0·0475573 grains or 0·003079 grammes. From this the respective weights of all the other numbers or degrees on this scale were calculated, on the principle that solids were to one another in the ratio of the cubes of their diameters. This mode of calculation is not, however, absolutely correct in principle, for the amount of flattening of the under surface of the globule diminishes in reality with the decreasing volume of the globule. In actual practice, however, this difference may be assumed to be so small that it may be neglected without injury to the correctness of the results.

The smaller the diameter of the globule, the less will be the difference or variation in weight in descending the degrees of this scale, since the globules themselves vary in weight with the cubes of their diameters; for this reason, also, all such globules as come within the scope of the balance employed should be weighed in preference to being measured, and this scale should be regarded as more specially applicable to the smaller globules beyond the reach of the balance.

| No. on scale | Greatest diameter in inches | Weight of globule in grains | Weight of globule in grammes |
|---|---|---|---|
| 1 | 0·0004 | 0·00000005 | 0·000000003 |
| 2 | 0·0008 | 0·00000044 | 0·000000028 |
| 3 | 0·0012 | 0·00000149 | 0·000000096 |
| 4 | 0·0016 | 0·00000355 | 0·000000229 |
| 5 | 0·0020 | 0·0000069 | 0·00000044 |
| 6 | 0·0024 | 0·0000119 | 0·00000077 |
| 7 | 0·0028 | 0·0000190 | 0·00000120 |
| 8 | 0·0032 | 0·0000284 | 0·00000184 |
| 9 | 0·0036 | 0·0000403 | 0·00000262 |
| 10 | 0·0040 | 0·0000554 | 0·00000359 |
| 11 | 0·0044 | 0·0000736 | 0·00000478 |
| 12 | 0·0048 | 0·0000958 | 0·00000620 |
| 13 | 0·0052 | 0·0001218 | 0·00000789 |
| 14 | 0·0056 | 0·0001522 | 0·00000985 |
| 15 | 0·0060 | 0·0001872 | 0·00001203 |
| 16 | 0·0064 | 0·0002272 | 0·00001471 |
| 17 | 0·0068 | 0·0002725 | 0·00001764 |
| 18 | 0·0072 | 0·0003234 | 0·00002094 |
| 19 | 0·0076 | 0·0003804 | 0·00002463 |
| 20 | 0·0080 | 0·0004437 | 0·00002872 |
| 21 | 0·0084 | 0·0005137 | 0·00003327 |
| 22 | 0·0088 | 0·0005906 | 0·00003823 |
| 23 | 0·0092 | 0·0006748 | 0·00004367 |
| 24 | 0·0096 | 0·0007668 | 0·00004964 |
| 25 | 0·0100 | 0·0008667 | 0·00005611 |
| 26 | 0·0104 | 0·0009749 | 0·00006311 |
| 27 | 0·0108 | 0·0010918 | 0·00007068 |
| 28 | 0·0112 | 0·0012176 | 0·00007883 |
| 29 | 0·0116 | 0·0013528 | 0·00008758 |
| 30 | 0·0120 | 0·0014976 | 0·00009696 |
| 31 | 0·0124 | 0·0016524 | 0·00010698 |
| 32 | 0·0128 | 0·0018176 | 0·00011677 |
| 33 | 0·0132 | 0·0019934 | 0·00012817 |
| 34 | 0·0136 | 0·0021801 | 0·00014114 |
| 35 | 0·0140 | 0·0023786 | 0·00015397 |
| 36 | 0·0144 | 0·0025879 | 0·00016755 |
| 37 | 0·0148 | 0·0028097 | 0·00018190 |
| 38 | 0·0152 | 0·0030437 | 0·00019705 |
| 39 | 0·0156 | 0·0032903 | 0·00021302 |
| 40 | 0·0160 | 0·0035550 | 0·00022983 |
| 41 | 0·0164 | 0·0038230 | 0·00024751 |
| 42 | 0·0168 | 0·0041096 | 0·00026606 |
| 43 | 0·0172 | 0·0044111 | 0·00028553 |
| 44 | 0·0176 | 0·0047250 | 0·00030589 |
| 45 | 0·0180 | 0·0050546 | 0·00032725 |
| 46 | 0·0184 | 0·0053991 | 0·00034955 |
| 47 | 0·0188 | 0·0057590 | 0·00037285 |
| 48 | 0·0192 | 0·0061344 | 0·00039716 |
| 49 | 0·0196 | 0·0065258 | 0·00042250 |
| 50 | 0·0200 | 0·0069335 | 0·00044890 |
| 51 | 0·0204 | 0·0073581 | 0·00047638 |
| 52 | 0·0208 | 0·0077799 | 0·00050495 |
| 53 | 0·0212 | 0·0082580 | 0·00053464 |
| 54 | 0·0216 | 0·00873438 | 0·00056549 |
| 55 | 0·0220 | 0·00922854 | 0·00059748 |
| 56 | 0·0224 | 0·0097412 | 0·00063067 |

| No. on scale | Greatest diameter in inches | Weight of globule in grains | Weight of globule in grammes |
|---|---|---|---|
| 57 | 0·0228 | 0·0102725 | 0·00066506 |
| 58 | 0·0232 | 0·0108228 | 0·00070021 |
| 59 | 0·0236 | 0·0113922 | 0·00073753 |
| 60 | 0·0240 | 0·0119815 | 0·00077570 |
| 61 | 0·0244 | 0·0125901 | 0·00081513 |
| 62 | 0·0248 | 0·0132119 | 0·00085588 |
| 63 | 0·0252 | 0·0138901 | 0·00089797 |
| 64 | 0·0256 | 0·0145440 | 0·00094141 |
| 65 | 0·0260 | 0·0152311 | 0·00098623 |
| 66 | 0·0264 | 0·0159472 | 0·00103245 |
| 67 | 0·0268 | 0·0166828 | 0·00108010 |
| 68 | 0·0272 | 0·0174414 | 0·00112918 |
| 69 | 0·0276 | 0·0182220 | 0·00117974 |
| 70 | 0·0280 | 0·0190256 | 0·00123177 |
| 71 | 0·0284 | 0·0198529 | 0·00128535 |
| 72 | 0·0288 | 0·0207035 | 0·00134041 |
| 73 | 0·0292 | 0·0215782 | 0·00139704 |
| 74 | 0·0296 | 0·0224469 | 0·00145525 |
| 75 | 0·0300 | 0·0234010 | 0·00151504 |
| 76 | 0·0304 | 0·0243496 | 0·00157645 |
| 77 | 0·0308 | 0·0253224 | 0·00163950 |
| 78 | 0·0312 | 0·0263228 | 0·00170422 |
| 79 | 0·0316 | 0·0273484 | 0·00177060 |
| 80 | 0·0320 | 0·0284000 | 0 00183869 |
| 81 | 0·0324 | 0·0294789 | 0·00190852 |
| 82 | 0·0328 | 0·0305838 | 0·00198008 |
| 83 | 0·0332 | 0·0317162 | 0·00205340 |
| 84 | 0·0336 | 0·0328768 | 0·00212851 |
| 85 | 0·0340 | 0·0340649 | 0·00220549 |
| 86 | 0·0344 | 0·0349739 | 0·00228400 |
| 87 | 0·0348 | 0·0364422 | 0·00235938 |
| 88 | 0·0352 | 0·0378008 | 0·00244730 |
| 89 | 0·0356 | 0·0390138 | 0·00253168 |
| 90 | 0·0360 | 0·0404368 | 0·00261797 |
| 91 | 0·0364 | 0·0417943 | 0·00270790 |
| 92 | 0·0368 | 0 0431930 | 0·00279642 |
| 93 | 0·0372 | 0·0446162 | 0·00288860 |
| 94 | 0·0376 | 0·0460718 | 0·00298279 |
| 95 | 0·0380 | 0·0475573 | 0·00307900 |
| 96 | 0·0384 | 0·0465239 | 0·00317728 |
| 97 | 0·0388 | 0·0506249 | 0·00327759 |
| 98 | 0·0392 | 0·0522069 | 0·00338020 |
| 99 | 0·0396 | 0·0538215 | 0·00348452 |
| 100 | 0·0400 | 0·0554688 | 0·00359138 |

*Cupellation Loss.*—This term is applied to indicate a minute loss of silver, unavoidably sustained in the process of cupellation, which arises from a small portion of that metal being mechanically carried along with the litharge into the body of the cupel. The amount of this loss increases with the quantity of lead present in the assay (whether contained originally in the assay or added subsequently for the purpose of slagging off the copper, &c.); it is relatively greater, as the silver globule is larger, but represents a larger percentage of the silver actually contained in the assay, in proportion as the silver globule obtained diminishes in size. It has, however, been experimentally proved that, in assays of like richness in silver, this loss remains constant when the same temperature has been employed, and similar weights of lead have been oxidised in the operation.

In the blowpipe assay this loss is not confined to the ultimate operation of cupellation, but occurs, though in a less degree, in the concentration of the silver-lead, and in the previous scorification of the assay, had such operation preceded the concentration. The total loss in the blowpipe assay is found, however, to be less than in the ordinary muffle assay, since in the latter case the whole of the oxidised lead is directly absorbed by the cupel.

In mercantile assays of ore it is not customary to pay attention to the cupellation loss, and the results are usually stated in the weight of silver actually obtained. Where, however, great accuracy is required, especially when the substances are very rich in silver, the cupellation loss is added to the weight of the silver globule obtained, in order to arrive at the true percentage.

The amount to be added for this purpose is shown in the annexed table, which is slightly modified from Plattner's.

The use of this table is best explained by an example, as the following:—An assay to which there had been added, in all, five times its weight of assay lead, gave a globule of silver equivalent to six per cent. Upon referring to the table, it will be seen that the cupellation loss for this would be 0·07; consequently the true percentage of silver contained in the assay would be 6·07. This table is only extended to whole numbers, but fractional parts can easily be calculated from the same.

When the globules of silver are so minute that they cannot be weighed, but must be measured upon the scale, the cupellation loss should not be added, since, as a rule, it would be less than the difference which might arise from

| Actual percentage of silver found by assay | Cupellation loss, or percentage of silver to be added to the actual percentage found by assay in order to show the true percentage of silver contained in same. The entire amount of lead in or added to the assay being the following multiples of the original weight of assay:— | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 8 | 11 | 13 | 16 |
| 99·75<br>99·5 . | 0·25 | 0·32 | 0·39 | 0·45 | 0·50 | | | | | |
| 90 . . | 0·22 | 0·29 | 0·36 | 0·42 | 0·47 | 0·69 | 0·83 | | | |
| 80 . . | 0·20 | 0·26 | 0·33 | 0·39 | 0·44 | 0·64 | 0·75 | | | |
| 70 . . | 0·18 | 0·23 | 0·29 | 0·35 | 0·40 | 0·58 | 0·68 | 0·82 | | |
| 60 . . | 0·16 | 0·20 | 0·26 | 0·30 | 0·36 | 0·52 | 0·61 | 0·74 | | |
| 50 . . | 0·14 | 0·17 | 0·23 | 0·26 | 0·32 | 0·46 | 0·54 | 0·65 | | |
| 40 . . | 0·12 | 0·15 | 0·20 | 0·22 | 0·27 | 0·39 | 0·46 | 0·55 | 0·62 | |
| 35 . . | 0·11 | 0·13 | 0·18 | 0·18 | 0·25 | 0·36 | 0·42 | 0·50 | 0·57 | |
| 30 . . | 0·10 | 0·12 | 0·16 | 0·16 | 0·22 | 0·32 | 0·38 | 0·45 | 0·51 | |
| 25 . . | 0·09 | 0·10 | 0·14 | 0·14 | 0·20 | 0·29 | 0·34 | 0·40 | 0·45 | |
| 20 . . | 0·08 | 0·09 | 0·12 | 0·12 | 0·17 | 0·25 | 0·29 | 0·35 | 0·39 | 0·45 |
| 15 . . | 0·07 | 0·08 | 0·10 | 0·11 | 0·15 | 0·20 | 0·23 | 0·28 | 0·32 | 0·37 |
| 12 . . | 0·06 | 0·07 | 0·09 | 0·10 | 0·13 | 0·17 | 0·19 | 0·23 | 0·26 | 0·32 |
| 10 . . | 0·05 | 0·06 | 0·08 | 0·09 | 0·11 | 0·15 | 0·17 | 0·20 | 0·23 | 0·27 |
| 9 . . | 0·04 | 0·05 | 0·07 | 0·08 | 0·10 | 0·14 | 0·16 | 0·18 | 0·21 | 0·25 |
| 8 . . | 0·03 | 0·04 | 0·06 | 0·07 | 0·09 | 0·13 | 0·15 | 0·16 | 0·18 | 0·22 |
| 7 . . | 0·02 | 0·03 | 0·05 | 0·06 | 0·08 | 0·12 | 0·13 | 0·14 | 0·16 | 0·20 |
| 6 . . | 0·01 | 0·02 | 0·04 | 0·05 | 0·07 | 0·10 | 0·11 | 0·12 | 0·14 | 0·17 |
| 5 . . | | 0·01 | 0·03 | 0·04 | 0·06 | 0·09 | 0·10 | 0·11 | 0·12 | 0·14 |
| 4 . . | | | 0·02 | 0·03 | 0·05 | 0·07 | 0·08 | 0·09 | 0·10 | 0·11 |
| 3 . . | | | 0·01 | 0·02 | 0·04 | 0·05 | 0·06 | 0·07 | 0·08 | 0·09 |
| 2 . . | | | | 0·01 | 0·03 | 0·04 | 0·04 | 0·05 | 0·06 | 0·07 |
| 1 . . | | | | | 0·01 | 0·03 | 0·03 | 0·04 | 0·04 | 0·05 |

errors of observation likely to occur when measuring their diameters upon the scale.

In the case of beginners, it will be found that the cupellation is usually carried on at too high a temperature, and that thereby a greater loss is occasioned than would be accounted for by the above table. After some trials the necessary experience will be acquired in keeping up the proper temperature at which this operation should be effected.

It now becomes necessary to consider in detail the processes requisite for extracting the silver contents (in combination with lead) from the various metallic alloys of silver which are met with in nature or produced in the arts.

In considering these, the following classification of the substances will be found convenient:—

I. METALLIC ALLOYS.
- *A. Capable of direct Cupellation.*
    - *a.* Consisting chiefly of lead or bismuth : silver-lead and argentiferous bismuth, native bismuthic silver.
    - *b.* Consisting chiefly of silver : native silver, bar silver, test silver, precipitated silver, retorted silver amalgam, standard silver, alloys of silver with gold and copper.
    - *c.* Consisting chiefly of copper : native copper, copper ingot, sheet or wire, cement copper, copper coins, copper-nickel alloys.
- *B Incapable of direct Cupellation.*
    - *a.* Containing much copper or nickel, with more or less sulphur, arsenic, zinc, &c.; unrefined or black copper, brass, german silver.
    - *b.* Containing tin : argentiferous tin, bronze, bell metal, gun metal, bronze coinage.
    - *c.* Containing antimony, tellurium, or zinc.
    - *d.* Containing mercury : amalgams.
    - *e.* Containing much iron : argentiferous steel, bears from smelting furnaces.

## A. METALLIC ALLOYS CAPABLE OF DIRECT CUPELLATION.

*a. Consisting chiefly of Lead or Bismuth.*—In determining the silver contained in these alloys, it is only requisite to place a clean piece of the same, weighing about from one to ten grains according to its probable richness in silver, upon a cupel of coarse bone ash, and proceed by concentration and cupellation exactly as has been already described under these heads.

Should the substance be not altogether metallic, or not free from adherent slag, earthy matter, or other extraneous matter, it should previously be fused on charcoal with a little borax in the reducing blowpipe flame, and the clean metallic globule then removed from the charcoal, and treated as before. In order to remove the globule from the inherent borax glass, it may be allowed to cool, and then detached ; or, after a little practice, it will be found easy, by a quick movement of the charcoal, to cause the globule, still melted, to detach itself completely, and drop on the anvil in the form of a single somewhat flattened globule, without suffering any loss of lead adhering to the charcoal.

In the case of argentiferous bismuth alloys the process is carried on in all respects the same as if silver-lead were being treated. As, however, the bismuth globule is very brittle, care must be taken when separating the concentrated globule from the litharge, as, if not carefully done, a loss may easily be sustained from a portion of the globule remaining behind adherent to the litharge. It is better, therefore, to remove the litharge by degrees from the globule with the aid of the forceps.

Argentiferous bismuth, free from lead, when cupelled alone, invariably leaves a globule of silver, having a dull frosted surface. If, however, at the end of the operation a small quantity of lead (¼ to ½ a grain) be added, and fused along with it, the silver globule then obtained will be perfectly bright and free from all bismuth.

In the case of native bismuthic silver it is advisable to fuse the previously weighed mineral with a little lead and borax glass on charcoal in the reducing flame, so as to free it from any adherent earthy matter, and then proceed by concentration and cupellation, as before described.

*b. Consisting chiefly of Silver: native silver, bar, test, and precipitated silver, retorted silver amalgam, standard silver, silver coin, and other alloys of silver with gold and copper.*— These alloys may be at once fused with lead on the cupel itself, and the operation finished as before described. In general, however, it is better to fuse the weighed assay previously with the requisite amount of pure lead and a little borax-glass, say from a quarter to half the weight of assay, in the reducing flame at a low heat on charcoal until the globule commences to rotate. This ensures the having a perfectly clean button of silver-lead, which is then cupelled in the ordinary manner.

In most cases the quantity of lead to be added need not exceed that of the weight of the alloy, but when several percentages of copper are present in the assay, as in case of many coins, &c., the lead should be increased to some three, or even five, times the weight of the assay in proportion to the amount of copper actually contained in the substance under examination, and which will be treated of more at length under the head of copper-silver alloys.

When no more lead has been added to the assay than its own weight, the cupellation may be concluded in one operation by inclining the stand, and so moving the globule on to a clean part of the cupel; but when more copper is present, it is preferable to concentrate first and cupel subsequently, in order thereby to reduce the cupellation loss to its minimum.

In the concentration as much copper as possible should be slagged off with the lead, which is effected by inclining the cupel somewhat more than usual, so that its surface may be less covered up with the litharge and exposed as much as possible to oxidation, by which means the litharge, as it forms, is enabled to carry off more of the copper contained in the silver-lead.

Should the silver globule after cupellation show indications of still containing copper, as before noticed, when treating of cupellation, a small quantity of lead must be fused along with it, and the cupellation finished as usual.

As at the present time no means are known by which silver can be separated from gold by the use of the blowpipe, in all cases of alloys containing gold, this metal remains to the last along with the silver, and the result in such cases always indicates the combined weight of both these metals contained in the alloy under examination. The employment of the humid assay must be resorted to for effecting their separation.

*c. Containing chiefly Copper: native copper, ingot, wire, or sheet copper, cement copper, copper coins, copper-nickel alloys.*—Under the most favourable conditions in cupellation, the amount of lead requisite, when converted into litharge, to slag of one part of copper along with it as oxide, amounts to between seventeen and eighteen times its weight. In the blowpipe assay it is usual to add to any cupriferous alloy an amount of pure lead equal to twenty times the amount of copper contained in the alloy, in order to ensure the whole of the copper being separated in the litharge. In the case of nickel the amount of lead required is somewhat less than with copper, but in practice the same amount of lead may be employed.

When the copper is quite clean the requisite amount of lead may be added to it in a single piece on the cupel, fused and cupelled as usual, after previous concentration of the silver-lead to a small-sized globule.

It is generally found, however, that traces of iron, slag, gangue, or other foreign matter, are present; and, consequently, it is usually advisable to fuse the assay along with the requisite amount of lead, and about one half its own weight of borax-glass in the reducing flame, until the whole of the substance is seen to have perfectly combined or alloyed with the lead, and the globule has entered into brisk rotation, whilst at the same time no detached metallic globules are seen in the borax-glass.

The concentration of the silver-lead and cupellation are then conducted as usual, taking care when concentrating to incline the cupel-stand so as to expose as much as possible of the metallic surface of the melted globule to the oxidising action of the air, with a view of enabling the litharge whilst forming to carry off as much copper along with it as possible.

Should the silver globule obtained after cupellation spread out, or appear to the eye more flattened than usual with globules of pure silver, it indicates that some copper still remains, and a small piece of assay lead (½ to 1 grain weight) should be placed alongside it whilst still on the cupel, fused together, and the cupellation finished on a clean part of the same cupel as usual.

Precipitated or cement copper, especially that which is in the crude state, and has not been melted and run into ingots, is often very impure, containing so much iron, lead, arsenic, earthy matter, &c., as not to admit of direct cupellation, and in such case should be treated as pertaining to class B. *a*.

### B. Metallic Alloys incapable of direct Cupellation.

*a. Containing much Copper or Nickel, with frequently some little sulphur, arsenic, zinc, iron, cobalt, &c., as unrefined or black copper, brass, German silver, &c.*—As the presence of these extraneous matters would interfere with the cupellation, either by causing a loss of silver-lead projected from the cupel upon the evolution of the volatile substances present, or by forming oxides which could not be absorbed by the cupel, it is necessary to eliminate such substances by a scorification with borax on charcoal, previous to concentration or cupellation.

In the case of unrefined and black copper, the portion used in the examination is placed in the scoop with twenty times its weight of assay lead, and its own weight of powdered borax-glass, mixed with the spatula, and transferred to a soda-paper cornet. It is then fused on charcoal in the reducing flame, which should be constant and uninterrupted, until all particles have completely united, and a brisk rotation sets in, which is kept up for a short time, when the silver-lead globule, which should appear bright on the surface after cooling, is concentrated and cupelled precisely as is directed under A. *c.* By this preliminary scorification the sulphur, arsenic, and zinc are volatilised, and any lead, cobalt, or iron slagged off into the borax-glass.

In the assay of brass and German silver, the quantity employed is fluxed with its own weight of borax-glass, but only requires ten times its weight of assay lead. The operation is commenced as before, but the globule is kept somewhat longer in rotation (always keeping the flame directed only on to the borax-glass), so as to allow the zinc present to be completely volatilised, which is evident when the surface of the silver-lead becomes bright, on which the heat is increased for a few moments to expel the last traces of that metal, and the silver-lead thus obtained is concentrated and cupelled as before.

The silver globule obtained from the cupellation of substances rich in copper generally requires the addition of a small quantity of lead and re-cupellation (as before described), in order to ensure its freedom from copper.

*b. Containing Tin: argentiferous tin, bronze, bell and gun metal, bronze coinage, &c.*—Alloys of silver with other metals containing tin do not admit of being cupelled, since the oxide of tin formed by the oxidation of that metal is not absorbed by the bone ash of the cupel along with the litharge; it consequently remains upon the surface of the cupel, and if present in any quantity interferes with the operation. As tin is not volatile when heated on charcoal, either in the oxidating or reducing blowpipe flame, it cannot be so dissipated, and in consequence, the entire amount of tin contained in any alloy under examination must be removed by oxidation or scorification from the silver-lead, previous to its being submitted to cupellation.

For this purpose, 1 part of the stanniferous alloy is fluxed with from 5 to 15 parts granulated assay lead (according to the amount of copper suspected to be present in the alloy), 0·5 part anhydrous carbonate of soda, and 0·5 part pulverised borax-glass, made up as usual in a soda-paper cornet, and the whole at first gently heated in reduction flame until the soda paper is charred and the alloy has afterwards united with the lead to form a single globule, whilst the borax and soda have combined as a glass or slag in which the soda prevents the easily oxidisable tin becoming oxidised to any extent before a perfect alloy has been formed with the lead, which then contains the whole of the silver.

As soon as this is effected, the blowpipe flame is altered to an oxidating one, and the metallic globule is kept at the point of the blue flame, which should touch it so as to cause the tin to become oxidised and be at once taken up by the glass surrounding it.

Should, however, it be seen that minute globules of metallic tin made their appearance on the outer edge of the slag or glass,* the operation must be at once discontinued, and the assay allowed to cool; after cooling the metallic globule is detached from the slag surrounding it, and being placed in a cavity on charcoal, is fused in the reducing flame along with a small piece of borax-glass and afterwards treated with the oxidating flame exactly as before (and if necessary, which is seldom the case, unless when treating argentiferous block tin, this operation may again require to be repeated), until it is seen that the surface of the metallic silver-lead globule does not any longer become covered with a crust or scales of oxide of tin, but presents a pure and bright metallic surface.

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* This occurs when the flux has become so saturated with oxide of tin that it cannot take up any more.

The silver-lead globule is now quite free from tin, and can be cupelled and the amount of silver determined as usual.

*c. Metallic alloys containing much antimony, tellurium, or zinc: antimonial silver and argentiferous antimony, telluric silver, and argentiferous zinc.*—Alloys of antimony with silver when treated on charcoal in the oxidating flame give off all their antimony, leaving the silver behind as a metallic globule having a frosted external appearance; telluric silver, on the contrary, however, when treated in a similar manner, only evolves a part of its tellurium, and even after cupellation with lead a small amount of tellurium generally remains behind alloyed with the silver.

All these compounds may be assayed as follows:—

One part of the alloy is placed in a soda-paper cornet along with 5 parts granulated assay lead, and 0·5 part pulverised borax-glass, and fused in reducing flame until the globule and slag are well developed; the oxidating flame is now directed on to the globule, causing the whole of the zinc, along with most of the antimony and part of the tellurium, to volatilise before the lead commences oxidising. The last traces of antimony are removed with some difficulty, during which operation some portion of the lead becomes oxidised. On cooling, the globule is now separated from the slag and concentrated upon a coarse bone-ash cupel as usual, and if no tellurium were present in the concentrated silver-lead, this may now be cupelled as usual.

If tellurium is present, as is seen by the concentrated globule of silver-lead possessing a dark-coloured exterior, it must be remelted with 5 parts assay lead and again concentrated; and these operations, if necessary, must be repeated until the surface of the concentrated globule is found to be clean and bright, as usual with pure silver-lead, when it may be cupelled fine and the silver globule weighed.

It sometimes happens, even after all these precautions have been taken, that the silver globule after cupellation shows a crystalline, greyish-white, frosted appearance, from its still containing tellurium; in such cases its own weight of assay lead (in one piece) should be placed beside it on the cupel, melted together, and the globule again cupelled fine on another part of the surface of the same cupel. In assaying substances very rich in tellurium the results obtained are, however, not very satisfactory, and may be as much as one or two per cent. too low, even after employing all precautions.

*d. Compounds of Silver with Mercury: arquerite, native and artificial amalgams and argentiferous mercury.*—The assay of these compounds is very simple. A weighed quantity of the liquid or solid amalgam is placed in a small bulb tube, and heated over the lamp very gradually in order to avoid spirting and to allow the mercury to volatilise quietly;* the heat is increased by degrees as long as any mercury is driven off, and the residue is heated for some time at a red heat in order to drive off as much mercury as possible without fusing the glass or causing the residual silver to adhere to it. The mercury expelled condenses itself above the bulb on to the upper part of the tube, and by gently tapping will collect in globules, which by carefully turning the tube, unite and can be poured out of the tube; after which the silver, left behind as a porous mass, may be removed from the tube, and after being fluxed with an equal weight of granulated assay lead and half its weight of borax-glass, must be fused on charcoal in the reducing flame, and the button, on cooling, cupelled as usual. Should, however, much copper have been present in the amalgam, a proportionately larger amount of assay lead is required to be added.

When the argentiferous residue is extremely small, as is often the case when assaying argentiferous mercury, this may adhere firmly to the glass of the tube. On such occasions this part of the tube must be cut off with the adherent residue, and the whole fused in a strong reducing flame along with its own weight of granulated assay lead, and with half its weight of anhydrous carbonate of soda. Upon cooling, the globule of silver-lead thus obtained is cupelled as usual.

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* In the case of solid amalgams, which often spirt very violently, this may be obviated by wrapping the assay in a small piece of tissue paper, and heating it in a blowpipe crucible, when all the mercury is given off quietly, leaving the silver behind.

*e. Compounds chiefly consisting of Iron: argentiferous-steel; cast-iron; bears from smelting furnace.*—Compounds consisting principally of iron with a small percentage of silver, although occasionally produced in the arts intentionally, as, for example, the so-called silver-steel, are commonly found on the blowing-out of furnaces used in the smelting of silver and copper ores, and are frequently rich in silver, as is the case with the bears from the silver furnaces at Kongsberg in Norway. An alloy of iron with silver is occasionally also found appearing in small quantities on the surface of melted silver in the process of casting, and in some cases at least this may be due to the action of the melted silver on the iron rods used for stirring up the molten metal.

As iron cannot be made to alloy itself with lead before the blowpipe, it becomes necessary to extract the silver by a more indirect process than is used in the case of other alloys containing that metal. In order to remove the iron the alloy must first be converted into sulphide of iron and silver, and to effect this the iron or steel must be reduced to powder, or fragments none greater than about a quarter of a grain in weight; for which purpose steel when hardened may require to be softened previously.

One part of the finely-divided iron or steel is now mixed with 0·75 part sulphur, eight parts granulated assay lead, and one part pulverised borax-glass; the mixture after being placed in a soda-paper cornet is carefully fused in a cavity on charcoal in the reducing flame, until the whole appears as a fluid globule containing both the lead and iron in combination with the sulphur. Without removing either this globule or the glass surrounding it from the charcoal, an amount of borax-glass in one or more fragments (in all about equal in weight to the original amount of iron employed), is now added (in order to combine with and slag off the whole of the iron), and fused along with the former globule, after which the whole is submitted to a strong oxidating flame until the impure lead globule shows itself protruding from the slag.

The charcoal is then inclined, so that the lead is alone subjected to the action of the outer flame, in order to volatilise the sulphur, and at the same time oxidise the iron which goes into the slag: this operation is continued until the globule of lead appears with a bright metallic surface; should it on cooling, however, be found to possess a black colour, and to be brittle, it must be still further oxidised as before described.

The silver-lead thus obtained will now be found to contain all the silver, and at the same time to be free from both iron and sulphur, and can be cupelled as usual.

No notice is here taken of alloys of silver and gold, since these metals cannot be separated before the blowpipe by any process yet known; and in all cases where gold may be present in an alloy, treated as here directed for obtaining its contents in silver, the gold also will be found to follow along with the silver, and must be parted from that metal by the humid method, in order to enable the true amount of silver present in the substance to be ascertained.

# CHAPTER XVI.

## THE ASSAY OF GOLD.

For the purposes of assay, all substances containing gold may be divided into two classes, as in the case of silver.

The *First Class* comprises all substances containing gold in a minute state of division; such, for instance, as those which, suitably pulverised, completely pass through a sieve of 80 holes to the linear inch. It often happens, however, that these substances contain fragments of gold of such magnitude as will not allow them to pass through the sieve: in such cases, that which passes *through* belongs to the first class, and that which remains *on* the sieve to the second class.

The *Second Class* comprises all alloys of gold, native or otherwise.

The name of substances belonging to this class is legion, for an extended examination shows that nearly every mineral substance contains more or less gold. The most common are—gold quartz, auriferous gossans, sulphides of iron (mundic), blende, copper pyrites, many antimonial minerals, galena, and nearly all the primitive rocks. All auriferous slags, amalgamation residues, and tailings, belong to this class.

*Assay of substances of the First Class.*—This assay is conducted in precisely the same manner as that of the corresponding silver class, which see. In case, however, the amount of gold present in the sample is small, as much as 2,000 grains, with flux suitably increased, may be employed. In case any metallic gold is left in the sieve, its amount is to be calculated as that of silver (see pages 476 and 477).

It may here be mentioned, that if silver or platinum coexist with the gold in the mineral subjected to assay, t will be found combined with the gold obtained by cupellation; and all gold so obtained must be submitted to the 'parting process,' which see under the head 'Assay of Auriferous Substances of the Second Class.' It may here be mentioned also, that the metallic gold left on the sieve must be thus operated on, as well as that obtained by fusion of the sieved ore and consequent cupellation, before the calculation given at pages 476 and 477 be entered into.

When gold is associated in quantity with quartz, its percentage can be approximatively ascertained in the same manner as that of pure tin-stone when mixed with quartz (see pages 414 and 415). If possible, a fragment of the gold must be detached from the quartz, and its specific gravity taken: if this be not possible, and the gold is nearly fine, the number 19 may be adopted. It is better, however, to determine experimentally the specific gravity of both quartz and gold.

### *Substances of the Second Class.*

Native gold.
Aurides of silver (native).
Gold and rhodium.
Gold and palladium.
Argentiferous telluride of gold.
Plumbo-argentiferous telluride of gold.
Sulpho-plumbiferous telluride of gold.
Artificial alloys of gold.

*Native Gold and Aurides of Silver* (*Native*), Au and $AuAg^n$, are found in variously contorted and branched filaments, in scales, in plates, in small irregular masses, in the crevices or on the surface of common ferruginous and other quartz. In Devonshire, at the Britannia Mine, it has occurred in pipes or veins, and disseminated in a compact hard gossan, one specimen of which was found to contain 27½ per cent. of fine gold; or, as in Wales, it largely accompanies blende and galena: it also occurs in a pyritous quartz; and it has been found in Scotland and Ireland. In the latter locality it occurred in the beds of streams as small scales and rolled masses, and nearly up to the present time this has been the most frequent mode of occurrence; but now, however, by the aid of improved machinery, rocks and minerals containing a comparatively small quantity can be profitably worked; and from this source, the greatest part of the gold poured into commerce is now extracted.

*Composition of several varieties of Native Gold, by Boussingault, the chief part from Central America.*

| | Malpaso | Llano | La Baja | Rio-Sucio |
|---|---|---|---|---|
| Gold . . | 88·24 | 88·58 | 88·15 | 87·94 |
| Silver . . | 11·76 | 11·42 | 11·85 | 12·06 |
| | 100·00 | 100·00 | 100·00 | 100·00 |

| | Ojas Anchas | Trinidad | Guano | Otramina |
|---|---|---|---|---|
| Gold . . | 84·50 | 82·40 | 73·68 | 73·60 |
| Silver . . | 15·50 | 17·60 | 26·32 | 26·60 |
| | 100·00 | 100·00 | 100·00 | 100·00 |

| | Titiribi | Marmato | Transylvania | Santa Rosa |
|---|---|---|---|---|
| Gold . . | 74·00 | 73·45 | 64·52 | 64·93 |
| Silver . . | 26·00 | 26·55 | 35·48 | 35·07 |
| | 100·00 | 100·00 | 100·00 | 100·00 |

*Specimens of Gold from Siberia, by Rose.*

| | Schabrowski, near Katherinenburg | Borushka, near Nischen-Tagil |
|---|---|---|
| Gold . . . . | 98·76 | 94·41 |
| Silver . . . . | 00·16 | 05·23 |
| Copper . . . . | 00·35 | 00·39 |
| Iron . . . . | 00·05 | 00 04 |
| | 99·32 | 100·07 |

| | Berescoff | Katherinenburg |
|---|---|---|
| Gold . . . . | 93·78 | 93·34 |
| Silver . . . . | 5·94 | 6·28 |
| Copper . . . . | ·08 | ·06 |
| Iron . . . . | ·00 | ·32 |
| | 99·80 | 100·00 |

| | Crascewo Nicolajensk, near Miask | Perroc Powlowsk, near Berescoff |
|---|---|---|
| Gold . . . . | 92·47 | 92·60 |
| Silver . . . . | 7·27 | 7·08 |
| Copper . . . . | ·06 | ·18 |
| Iron . . . . | ·08 | ·06 |
| | 99·88 | 99·92 |

| | Borushklei | Alexander Andrejensk, near Miask |
|---|---|---|
| Gold . . . . | 90·76 | 87·40 |
| Silver . . . . | 9·02 | 12·07 |
| Copper . . . . | ·09 | ·09 |
| | 99·87 | 99·56 |

### *Gold from Senegal, by D'Arcet.*

| | |
|---|---|
| Gold | 86·97 |
| Silver | 10·53 |
| | 97·50 |

### *Gold from Brazil, by D'Arcet.*

| | |
|---|---|
| Gold | 94·00 |
| Silver | 5·85 |
| | 99·85 |

### *Gold from Anamaboe, Africa, by Henry.*

| | |
|---|---|
| Gold | 98·06 |
| Silver | 1·39 |
| Iron | ·15 |
| | 99·60 |

### *Gold from California, by Henry.*

| | 1. | 2. |
|---|---|---|
| Gold | 86·87 | 88·75 |
| Silver | 12·33 | 8·88 |
| Copper | ·29 | ·85 |
| Iron | ·54 | traces |
| Silica | ·00 | 1·40 |
| | 100·03 | 99·88 |

### *Gold from California, by Teschemacher.*

| | |
|---|---|
| Gold | 90·33 |
| Silver | 6·80 |
| Oxide of iron | 1·10 |
| Sand | ·66 |
| | 98·89 |

### *Gold from Australia, by Henry.*

| | |
|---|---|
| Gold | 95·68 |
| Silver | 3·92 |
| Iron | ·16 |
| | 99·76 |

### *Gold from Devonshire and Wales by the Author.*

The author has received two specimens of gold, one from Wales, and the other from the Britannia Mine, Devon; and found both to be absolutely fine gold.

*Gold and Rhodium.*—This compound was discovered by M. André del Rio among some gold ores in Mexico. It has a gold colour, and contains variable proportions of rhodium; the mean, however, is 34 per cent.

*Gold and Palladium.*—The following is the composition of this alloy :—

| | |
|---|---|
| Gold | 85·98 |
| Palladium | 9·85 |
| Silver | 4.17 |
| | 100·00 |

*Argentiferous Telluride of Gold* ($AgTe_2 + 3AuTe_6$).
Composition :—

| | |
|---|---|
| Gold | 30 |
| Silver | 10 |
| Tellurium | 16 |
| | 56 |

*Plumbo-argentiferous Telluride of Gold* (probable formula, $AgTe_2 + 3AuTe_3 + 2PbTe_2$).
Composition :—

| | |
|---|---|
| Gold | 26·75 |
| Silver | 8·50 |
| Lead | 19·50 |
| Tellurium | 44·75 |
| Sulphur | ·50 |
| | 100·00 |

*Sulpho-plumbiferous Telluride of Gold* (probable formula $AuTe_3 + 4PbTe_2 + 2PbS$).
Composition :—

| | |
|---|---|
| Gold | 9·0 |
| Silver | ·5 |
| Lead | 54·0 |
| Copper | 1·3 |
| Tellurium | 32·2 |
| Sulphur | 3·0 |
| | 100·0 |

*Artificial Alloys of Gold.*—The only one of these alloys which will be specially noticed here, is the standard gold of this realm. It is composed of 22 parts of fine gold and 2 parts of alloy (copper), constituting 22 carat or standard gold.

### *General Observations on the Assay of Gold Alloys.*

*Cupellation, Gold and Lead.*—The cupellation of the alloys of gold and lead is conducted in a similar manner to those of silver and lead. It presents even less difficulty, and requires less precaution, because it is not so volatile, and because it has a less tendency than silver to penetrate into the cupel, and the button is less subject to throw pieces out of the cupel. These cupellations take place at a higher temperature than those of silver, and we need not be afraid of giving a good heat at the moment of brightening: the gold is but the purer.

Mr. Makins made the following statements on certain sources of loss of precious metals in some operations of assaying before the Chemical Society, January 19, 1860. When making a large number of assays of gold, and also estimating the silver, under circumstances which required that an extraordinary degree of heat should be employed, Mr. Makin was struck with the great loss of gold and silver. Satisfied that it was not entirely owing to 'cupel absorption' he examined the contents of the iron flue of the furnace, which had only been used for the cupellation of gold assays, to see if any of the precious metals had been volatilised. Under the microscope the apparently carbonaceous matter from the flue showed yellow masses of oxide of lead, nodules of suboxide of copper, and minute grains of silver mixed with carbonised matter containing small grains of unburnt fuel. On analysis the metallic matters present were found to be oxide of lead mixed with small portions of gold, silver, and oxide of copper. The metals were extracted by lead in the usual way, and the button obtained by scorification was subjected to cupellation. The gold and silver were then parted, and the proportions of each in 1,000 grains were found to be:—

| | |
|---|---|
| Gold . . . . . . . . . | 0·087 |
| Silver . . . . . . . . . | 0·763 |

*Gold and Copper, proportion of Lead.*—The alloys of gold and copper are cupelled like the alloys of gold and silver; but as copper has a very great affinity for gold, it is necessary to use a larger proportion of lead to ensure its oxidation when combined with gold than when united with silver. This proportion varies according to the standard and the temperature. It is admitted that for the same standard there must, under similar circumstances, be twice as much lead used in the cupellation of gold as for that of silver. Thus, 14 parts, at least, ought to be employed in common furnaces for an assay of gold coin which contains 0·1 of copper. There is no inconvenience in employing a little more, as it does not increase the loss of gold. However great the proportion of lead may be that is added to the cupreous gold for the purpose of cupellation, the button retains always a very small quantity of copper, which a fresh cupellation does not free it from, and which occasions what is termed the surcharge. This surcharge being very slight, can be neglected in assays of minerals; but it is necessary to take notice of it in the assay of alloys. But it is known that the presence of silver much facilitates the separation of copper from gold, and it is rare that an alloy of cupreous gold does not contain a little silver, which must be separated: and when that is not the case, a small quantity of that metal can be introduced into the alloy, so as to be in about the proportion of 3 parts to 1 of gold. When an assay is to be made of an alloy of gold and copper, a sufficient quantity of silver is to be added to fulfil this condition according to the presumed standard, which is determined approximatively by a preliminary assay, and then cupelled with lead.

The examination on the *touch-stone* is based upon the fact, that the richer an alloy is in gold the more clearly does a streak drawn with it on a black ground present a pure gold-yellow colour, and the less is it attacked by pure nitric acid or by a test acid. This *test acid* consists of ninety-eight parts pure nitric acid of 1·34 spec. grav. (37° Beaumé), two parts pure hydrochloric acid of 1·173 spec. grav. (21° B.), and twenty-five parts distilled water. To judge of the richness of the alloy to be examined, its streak is compared with marks drawn with alloys (*the touch-needles*) whose richness is accurately known. In order to get correctly the streak of the alloy to be tested, the surface of the metal must first be somewhat filed away, since this may be impure, or, as with coins and jewelry, it may have been made somewhat richer by boiling with acid, and the so-called colouring of the goldsmith, and a clean fracture is rarely to be obtained. Five series of prepared touch-needles are required. The first series consists of copper and gold, and is called the *red series*, and the proportion of gold increases by half carats in the successive needles. The second series, the *white series*, contains needles of gold and silver, in which the proportion of gold likewise increases by half carats. The third series, a *mixed one*, contains needles in which the quantities of silver and copper are equal, and the proportion of gold also increases by half carats. The fourth consists also of needles for a mixed series, in which the silver is to the copper as 2 : 1, and the gold increases by half carats; and the fifth is also formed of needles for a mixed series, in which the quantity of silver is to that of the copper as 1 : 2. Moreover, in mints and stamping bureaux, alloys are used which correspond precisely to the legal standards. The testing upon the touchstone begins by determining to which series the alloy to be examined belongs. Then those touch-needles are rubbed against the stone whose marks most nearly approximate in colour to that of the alloy. The marks must form a thin continuous layer. A drop of pure nitric acid is now placed upon them with a glass rod, and its comparative effect observed. The acid is allowed to work a short time, and then wiped off, in order to see whether the streak appears unchanged, or whether it has more or less disappeared. The test acid above is also used. This is so composed that it does not work at all upon an alloy containing eighteen carats and more of gold, and with such an alloy the streak, after using the acid, will not be wiped off with a fine linen rag, provided that stone and acid had a temperature of 10 to 12° C. Pure nitric acid produces almost no effect upon an alloy of fifteen or sixteen carats fine, and over. The testing on the touchstone can indeed make no pretension to accuracy, especially where the amount of gold is small, but it yields sufficiently useful results for a preliminary test. It requires, however, a sharp and very practised eye. Moreover, the preparation of the touch-needles is wearisome, as the required proportion is not always quickly reached, nor are good malleable alloys always obtained. The touchstone, therefore, is in general only used where frequent gold assays are to be made of alloys varying in richness, or where (as frequently with gold plate) an examination on the touchstone will suffice.

TABLE FOR PROPORTION OF LEAD TO BE EMPLOYED IN THE CUPELLATION OF GOLD AND COPPER.

| Gold in Alloy | Lead required | Ratio of lead in the assay to the copper, &c. | |
|---|---|---|---|
| 1000 thousandths | 1 part | —— | |
| 900 „ | 10 parts | 100,000 : 1 | |
| 800 „ | 16 „ | 80,000 : 1 | |
| 700 „ | 22 „ | 73,333 : 1 | |
| 600 „ | 24 „ | 60,000 : 1 | |
| 500 „ | 26 „ | 52,[illegible]00 : 1 | 0 |
| 400 „ | 34 „ | 56,666 : 1 | |
| 300 | | 48,571 : 1 | |
| 200 | | 42,500 : 1 | |
| 100 | | 37,777 : 1 | |
| 50 | | | |

Kandelhardt gives the ratio in the following table :—

| Gold in 1000 parts | Quantity of lead required |
|---|---|
| 1000 fine gold | 8 times the weight of the alloy |
| 980 — 920 | 12 „ „ „ |
| 920 — 875 | 16 „ „ „ |
| 875 — 750 | 20 „ „ „ |
| 750 — 600 | 24 „ „ „ |
| 600 — 350 | 28 „ „ „ |
| 350 — 0 | 32 „ „ „ |

*Gold, Silver, Platinum, and Copper.*—The presence of platinum in an alloy renders the separation of the oxidisable metals, more especially copper, very difficult by cupellation. It appears, indeed, that it would be almost impossible to arrive at it, if the alloy of copper contained nothing but gold and platinum. It is necessary that silver be present at the same time. When this metal is absent, it is requisite to add a quantity of it, which ought to be equivalent to double the weight of the gold and platinum united, and cupel at the strongest heat which can be obtained in a good muffle with a suitable proportion of lead. This proportion varies much according to the composition of the alloy, and the temperature at which the operation is carried on.

Experience has shown that the copper can be more completely separated, and less silver lost, by cupelling at a high temperature, with the least possible quantity of lead, than by employing more lead, and working at a lower temperature. M. Chaudet has made several assays, in order to determine the proportion of lead required for the cupellation of the three following alloys:—

| | 1. | 2. | 3. |
|---|---|---|---|
| Gold | 0·100 | 0·020 | 0·005 |
| Platinum | 0·100 | 0·200 | 0·300 |
| Silver | 0·250 | 0·580 | 0·595 |
| Copper | 0·550 | 0·200 | 0·100 |

And has found, for the first, that by employing 20 parts of lead the separation is very nearly complete; but that at a higher temperature there is a loss of silver; and in order to render the assay correct, it must be cupelled at the latter temperature, with only 14 of lead; for the second, 8 of lead, at a high temperature; and for the third 30 parts of lead are necessary, at the same high temperature of the muffle; but it is almost impossible to separate all the copper, and no advantage can be obtained by increasing the quantity of lead. When almost the last traces of the copper are separated, the button must be cupelled afresh, with a small quantity of lead; but a small quantity of silver is nearly always lost. In all cases, in order that no lead shall remain, it is necessary to leave the assay button some few minutes in the muffle, after cupellation is finished.

The alloys of gold and silver which contain platinum show, either by cupellation or parting, certain characters which prove the presence of that metal. If the assay be not heated very strongly, it does not pass, and the button becomes flat: this effect becomes very sensible when the platinum is to the gold as the proportion of 2 to 100. Under the same circumstances, the nitric acid solution proceeding from the parting is coloured straw-yellow. At the moment an assay of an alloy containing platinum terminates, the motion is slower, and the coloured bands are less numerous, more obscure, and remain a much longer time than when there is no platinum: the button does not uncover, and the surface does not become as brilliant as that of an alloy of gold or silver, but it remains dull and tarnished. When the assay is well made, it is to be remarked that the edges of the button are thicker and more rounded than in ordinary assays, and it is of a dull white, approaching a little to the yellow; and lastly, its surface is wholly or in part crystalline. These effects are sensible even when the gold does not contain more than 0·01 of platinum. When the alloy contains more than 10 parts of platinum to 90 of gold, the annealed cornet produced in the parting process is of a pale yellow, or tarnished silver colour.

*Gold alloyed with Silver.*—The separation of gold from silver is termed *parting*. Parting is not only used to separate silver from gold, but for the separation of other metals, such as copper, when cupellation does not separate it entirely. Parting by the wet process is carried on by the means of nitric acid, *aqua regia*, or sulphuric acid.

When an alloy of gold and silver has been reduced by a flatting mill to very thin plates, it is sufficient that it contains 2½ of silver to 1 of gold in order that the parting may be effected completely by nitric acid, and takes place much less easily when the silver in the alloy is in larger proportion: but when this proportion exceeds 3 parts of silver for 1 of gold, then the latter is obtained in leaves so fine, that there is risk incurred of losing some in the subsequent manipulation, and even by the act of boiling the acid liquid.

We must always, therefore, when a very exact assay is required, contrive that the alloy shall contain a little less than 3 parts of silver to 1 of gold; a proportion which long experience has demonstrated to be the best. If the alloy contain less than 2½ of silver to 1 of gold, the silver does not wholly dissolve, because there is a part of it so enveloped in the gold that the strongest acid does not act on it.*

*Inquartation.*—The operation by which the alloy is brought to this standard is termed *quartation*, or *inquaration*. It consists in fusing the alloy in a cupel, with 2 parts of lead and the quantity of fine silver, or fine gold, necessary to bring it to the desired composition. This quantity is estimated according to the approximative determination of the standard of the alloy, which ought to be made either by means of a preliminary assay, as hereafter described, or by means of the touchstone. If we do not employ the whole of the alloy the assay will not be exact, because the gold and silver are not always found distributed in an uniform manner; at least, every time it is not poured into a cold ingot mould.

---

* **Pettenkoffer and others have shown that less than two parts of silver will suffice, and be even advantageous.**

*Operation.*—The cupelled and quartated button is flattened on an anvil and annealed, in order to soften it. It is laminated to give it a certain thickness, and is then annealed afresh, and rolled into a cornet or spiral around the quill of a pen. It is necessary that the alloy should be reduced to a suitable thickness, on the one hand, in order that the silver may be dissolved completely: and, on the other, that the plate of gold may remain whole after the operation. The following is that which experience has proved best. The quantity of matter operated upon, or taken for the assay, should be about 12 grains; and the alloys resulting from these 12 grains, and the silver, employed in the inquartation into a plate of from 18 to 20 lines in length and 4 or 5 in breadth.

The cornet for assay is placed in a glass matrass, capable of containing about three ounces of water; pure nitric acid is added at different times, and heat applied. When all the silver is dissolved, it is washed by decantation with water; the matrass is reversed into a small crucible, the cornet falls out and is dried. In this state the cornet is very fragile, and of a dull red colour; it is annealed in a muffle, and heated gradually without fusion. It becomes thereby much contracted, and acquires a metallic lustre, and so much solidity that it can be weighed without fear of breaking it. Its weight can be ascertained in the assay balance.

There are many ways of employing nitric acid. Formerly 2½ ounces (thirty-five times the weight of the alloy) of nitric acid (1·15 sp. gr.) was poured upon the inquartated cornet, and boiled gently for fifteen or twenty minutes, the liquid decanted and replaced by 1½ of acid (1·24 or 1·26), twenty-four times the weight of the alloy, boiling for twelve minutes, then decanting and washing, &c. Vauquelain advised, in his 'Manuel de l'Essayeur,' to pour on the quartated cornet—the weight of the assay being 7·7 grains—554 to 770 grains of nitric acid (1·16 sp. gr.), which ought to fill the matrass half or two-thirds, and boil gently for twenty, or twenty-two minutes at most, to decant and replace the liquid by 500 to 800 grains of acid (1·26 sp. gr.), and to boil for eight or ten minutes. The assay is to be acted on always twice, because, if we employ at once very strong acid, the action will be too brisk, and the cornet might be broken or carried out of the matrass, and, on the other side, the acid of 1·16 sp. gr. cannot dissolve the last portions of silver, which are very difficult to separate from the gold.

*Surcharge.*—It is remarked that by following this method the cornet always retains a small quantity of silver, so that fine gold submitted to quartation and parting always weighs more after than before the operation. The augmentation of weight which it undergoes is termed the *surcharge*; this surcharge is commonly from 0·001 to 0·002. M. Chaudet has found means to avoid it. In order to do so, pour on to the quartrated cornet nitric acid of 1·16 sp. gr., and heat for three or four minutes only; replace this acid by acid at 1·26 sp. gr., and boil during ten minutes; decant and make a second boiling with acid at 1·26 sp. gr., which boil for eight or ten minutes. The assay requires but from twenty to twenty-three minutes, and, according to M. Chaudet, gives perfectly pure gold.

The following statements referring to this subject were made in the Chemical Society on January 19, 1860:—

A source of loss occurs in parting operations and refining on the large scale, from the solution of gold in nitric acid, even when it is quite free from hydrochloric acid, in consequence of the formation of nitrous acid. Mr. Field, the Queen's assay master, had a bottle which was thickly coated with gold deposited from nitric acid which had been used in parting operations. Sir John Herschel thought that the gold might have been suspended in the acid; but the fact that the bottle was pear-shaped, and was uniformly coated with the metal, proved that it must have separated from a solution. To ascertain the amount of loss from this source in ordinary assay operations, Mr. Makin took four specimens of pure gold accurately weighed, added the usual proportions of fine silver and lead, and then cupelled them. The resulting buttons were rolled, coiled, and parted with nitric acid, the cornets being boiled in two acids of different strengths a different number of times. Calling the weighings before the operation 1000, the results were as follows :—

| | | |
|---|---|---|
| 1. | Boiled in acid twice | 999·6 |
| 2. | „ three times | 999·2 |
| 3. | „ four „ | 998·7 |
| 4. | „ five „ | 997·9 |

The loss is thus seen to increase as the boilings are multiplied.

When silver is present in large quantity, Mr. Makin believes that the solvent action of nitrous acid is restrained by electrical action, the gold becoming the negative and the silver the positive pole of a circuit; but as the silver is removed, the solution of the gold goes on more rapidly. The cause of the evolution of nitrous acid is evident as long as there is any silver present, and it often results from the use of charcoal to prevent 'bumping.' When charcoal is thoroughly carbonised, it does not materially affect the acid; but if it contain woody matter, nitrous acid is sure to be set free. Mr. Makin has given up the use of charcoal on this account.

The commercial importance of this subject will be admitted, when we remember the enormous value of the metals dealt with in this country, and that the question of profit and loss in commercial transactions with them are almost entirely in the hands of the assayer. A knowledge of these facts may also serve to account for some of the discrepancies between assayers.

*Argentiferous and Auriferous Ores.*—In the assay of auriferous ores, the button produced by cupellation commonly contains silver. When the proportion of this metal surpasses that of inquartation, the button is flattened between two pieces of paper, and treated by pure nitric acid.

The gold remains under the form of a yellowish-brown powder, which is weighed immediately, or fused in the cupel enveloped in a sheet of lead. When the quantity is extremely small and imponderable, we can assure ourselves at least of its presence by treating the residue left by nitric acid with *aqua regia*; if it contain gold, it dissolves and gives a yellowish liquid, in which a drop of solution of chloride of tin or the crystallised chloride forms a deposit of purple of Cassius of a violet colour: this character proves the presence of the smallest traces of gold. When the gold predominates in the button, it is necessary to re-fuse it with three times or less its weight of silver, and recommence the assay with the addition of this preparation of silver.

*Sea-salt.*—We can, according to M. Gay-Lussac, make assays of the alloys of gold, silver, and copper with great exactitude, by means of the standard solution of sea-salt. When the alloy contains five or six times more of silver and copper than of gold, a known weight of the alloy is taken, containing nearly 1 gramme of silver; it is dissolved in a matrass (fig. 128) capable of containing about 200 grammes of water, with 462 grs. of nitric acid, at 1·26 sp. gr., and boiled for ten minutes. The assay is finished as usual; but in order to leave the gold and separate the silver, supersaturate the solution with ammonia, which dissolves the chloride; wash the residue twice in succession with ammonia, then place in a crucible to anneal. If the gold were alloyed with silver and copper in a larger proportion than 1 to 6, a known quantity of fine silver should be added, and then deducted from the assay. In order to avoid all loss, the bottom of the crucible is lined with paper, and the alloy placed thereon, and the latter covered with fused borax.

Fig. 128.

M. J. Nicklès, in some remarks on the extraction of auriferous silver from its ores, says, that 'though the treatment of argentiferous ores is easy, and that of auriferous ores not very complicated, it is otherwise when the two metals are associated, for then the properties of the one prevent the manifestation of the properties of the other. If, for instance, auriferous silver is treated by chlorine water, the core immediately becomes covered with a coating of chloride of silver, which protects the rest from the action of the solvent. If this is attacked by salt water, ammonia, or hyposulphite of soda, the core becomes unmanageable, the chloride of silver dissolves, it is true, but leaves behind it a layer of metallic gold which in its turn resists the action of the solvents of chloride of silver.

'After many tentative trials the simple plan occurred to the author of associating the two solvents, chlorine and chloride of sodium. He took salt water concentrated and saturated with chlorine, and digested the auriferous alloy in it. By burning an ore of this kind and then washing it with the above solvent, the chlorine attacks the metallic particles, and then transforms them into chloride, which is dissolved by the sea-salt.

'It is thought that this solvent may serve for the treatment of ores so poor in metals as to be discarded for the ordinary extracting processes.'

*Aqua Regia.*—When gold is the largest portion of the alloy, and when there are reasons for not adding silver, the parting can be made by *aqua regia.* In this case, all the gold is dissolved, and the silver converted into chloride; the chloride is washed, dried perfectly, and weighed. When the gold is precipitated by proto-sulphate of iron, it is washed with a little muriatic acid, and annealed strongly before weighing or even carrying the annealing so far as to fuse it, and then cupelling it with lead.

If an alloy, containing much silver, be treated by this process, it sometimes happens that the excess of chloride of silver prevents the complete solution of the gold. In this case it is necessary to reduce the alloy to an excessively thin plate, to dissolve the chloride in ammonia, and to treat afresh with *aqua regia.* This process can rarely be made use of in the large scale, because the precipitation of gold by sulphate of iron is long and troublesome.

*Method of M. Rose.*—M. G. Rose fuses the alloy with lead, over a spirit-lamp, in a porcelain crucible, acts on it with nitric acid, which dissolves the silver and lead, precipitates the silver by a solution of chloride of lead; lastly, the auriferous residue is dissolved by *aqua regia*, and the gold precipitated by protochloride of iron.

*Standard of the Alloys of Gold.*—The real standard of the alloys of gold is expressed in fractions of unity, as in the case of alloys of silver. We suppose 24 carats in unity, and 32-32nds in the carat; the unity contains then 768-32nds. After these data the following table has been formed, which expresses the relation of 32nds and carats to decimal fractions of the unity.

| 32nds | Decimals | Carats | Decimals |
|---|---|---|---|
| 1 | 0·001302 | 1 | 0·041667 |
| 2 | 0·002604 | 2 | 0·083334 |
| 3 | 0·003906 | 3 | 0·125001 |
| 4 | 0·005208 | 4 | 0·166667 |
| 5 | 0·006510 | 5 | 0·208333 |
| 6 | 0·007912 | 6 | 0·250000 |
| 7 | 0·009115 | 7 | 0·291666 |
| 8 | 0·010415 | 8 | 0·333333 |
| 9 | 0·011718 | 9 | 0·374999 |
| 10 | 0·013021 | 10 | 0·416667 |
| 11 | 0·014323 | 11 | 0·458630 |
| 12 | 0·015625 | 12 | 0·500000 |
| 13 | 0·016927 | 13 | 0·541667 |
| 14 | 0·018230 | 14 | 0·583333 |
| 15 | 0·019531 | 15 | 0·624555 |
| 16 | 0·020833 | 16 | 0·666667 |
| 17 | 0·022135 | 17 | 0·707333 |
| 18 | 0·023436 | 18 | 0·750000 |
| 19 | 0·024740 | 19 | 0·791666 |
| 20 | 0·026042 | 20 | 0·833333 |
| 21 | 0·027343 | 21 | 0·874999 |
| 22 | 0·028644 | 22 | 0·916666 |
| 23 | 0·029948 | 23 | 0·958333 |
| 24 | 0·031250 | 24 | 1·000000 |
| 25 | 0·032552 | | |
| 26 | 0·033854 | | |
| 27 | 0·035156 | | |
| 28 | 0·036460 | | |
| 29 | 0·037760 | | |
| 30 | 0·039062 | | |
| 31 | 0·040364 | | |
| 32 | 0·041667 | | |

## *Assay of the Alloys of Gold and Copper, or Gold, Silver, and Copper.*

*Preliminary Assay.*—As in the case of silver assaying the quantity of lead to be employed is of importance, a preliminary assay must be made when the standard of the alloy to be examined is not approximatively known. It is thus effected:—To 2 grains of the alloy add 6 grains of fine silver and 50 grains of pure lead. The lead must be introduced into a hot cupel, and when fused, and its surface fully uncovered, the alloy and silver may be added, wrapped either in thin paper or a small quantity of lead foil. The cupellation finished, and the cupel cold, the button of gold and silver must be removed from the cupel by aid of the pliers, and if necessary cleansed. Hammer it to a thin plate on the anvil, place it in a small evaporating basin, and treat it with half an ounce of nitric acid. (It may be here mentioned, that the nitric acid employed in the assay of gold must be chemically pure, and special care must be taken that it contains no trace of chlorine.) The evaporating basin is gently heated until all action ceases. The brownish residue is repeatedly washed with hot water, dried, ignited, and weighed; and from its weight the amount of lead and silver to be added in the actual assay may be determined. The presence of copper in the alloy is indicated by the blackness of the cupel where it is saturated with oxide.

*Assay Proper.*—In this case it will be supposed that standard gold is the alloy operated on, and that preliminary assay has given about 91½ per cent. of gold. On referring to the table (page 610), it will be found that between 27 and 30 parts of lead are required for such a percentage of gold, and that, according to the general observations on this class of assay, three times its weight (that is, the weight of fine silver) will be required to so dilute the gold that nitric acid can attack and dissolve out the whole of the silver combined with it.

Place the weight representing 24 carats in the pan of the balance, and exactly counterpoise it with the gold to be assayed; two portions should be thus weighed. Two portions of fine silver must now be weighed; 33 grains will be required for each 24 carats of gold, as 22 carats, or 11 grains, of fine gold exist in the 24 carats, and three times the quantity of silver is necessary. 300 grains of lead must be placed in a hot cupel (two being thus prepared), and, as in the preliminary assay, when the surface is fully uncovered, the gold and silver are added, and the cupellation proceeded with, taking all the precautions already fully pointed out elsewhere.

The button so obtained is cleansed, hammered on the anvil, then annealed and passed between the rollers of a small flatting-mill; being occasionally annealed, in order to prevent the laminated button cracking at the edges. When reduced to the desired degree of thinness it is again annealed, and rolled round a quill or glass rod into a spiral, termed a cornet. This cornet is placed in a parting flask with 1½ oz. of nitric acid, sp. gr. 1·16, very gently heated to the boiling-point, and at that maintained for ten minutes. The acid is then to be poured off, and 2 oz. of nitric acid, sp. gr. 1·26, added, and again boiled for ten minutes. This second acid is also poured off, and a third quantity of like specific gravity added and boiled. The cornet is then well washed with distilled water, and the flask, filled with distilled water, is inverted, having its mouth closed with the thumb. The cornet will fall through the water without breaking, and can be introduced, together with some of the water, into a small crucible (cornet crucible), the water poured off, the crucible and gold gradually dried, and then heated to redness. When cold, the final operation of weighing may be performed, thus:—The weight representing 22 carats is placed in one pan of the balance, and the cornet in the other: as the gold employed was supposed to be standard, it ought to weigh exactly 22 carats. If, however, gold of greater or less fineness had been submitted to assay—say of 23 and 21 carats respectively—1 carat weight would have been required in the pan containing the 22 carat weight, to counterbalance the gold carat: in this case the gold would be 23 carats fine, or, in the usual mode of reporting, 'one carat better.' If, on the other hand, the 1 carat weight had been found necessary in the pan containing the cornet, the gold would be 21 carats fine, or 'one carat worse.'

In cases where it is known that the gold under examination contains no silver, the only alloy being copper, its fineness can be determined by cupelling 24 carats with its proper portion of lead, and weighing the resulting button, which should represent the amount of fine gold in the alloy assayed.

*Parting Assays.*—Parting assays are those assays by which the amount of fine gold and fine silver in any alloy is determined. When the amount of gold exceeds that of the silver, it is called 'gold parting;' when the amount of silver exceeds that of the gold, 'silver parting.'

In this assay the weights employed in the silver assay are employed, as the report is made in ounces of fine metal per pound Troy.

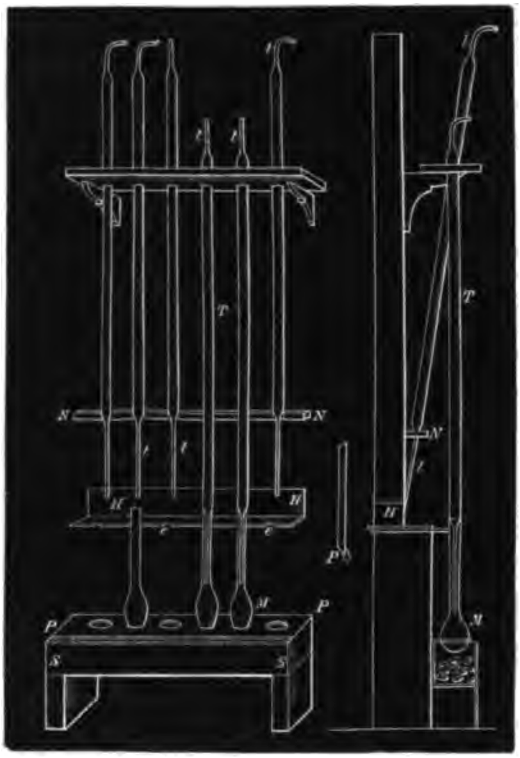
12 grains (representing 1 lb. Troy) of the alloy are weighed off, cupelled with 300 grains of lead, and the resulting button, containing only gold and silver, is weighed. Suppose it weigh 10 grains, then 2 grains, = 2 ounces in the pound of alloy, is copper or some other metal, which has been oxidised and carried into the cupel with the litharge. A preliminary assay must be made of the alloy, to ascertain the approximative quantity of silver and gold, so as to apportion the amount of silver in the assay proper: this amount being found, it is to be weighed off, added to the button of fine gold and silver obtained as above, and the whole cupelled with 200 grains of lead; the cupelled mass of gold and silver laminated and treated with nitric acid, as already described, and the resulting gold weighed. Suppose the weight to be 8 grains, = 8 ounces, the result would stand thus:—

| | |
|---|---|
| Copper or other base metal . . . . | 2 oz. |
| Gold . . . . . . . . . | 8 „ |
| Silver . . . . . . . . . | 2 „ |
| | 12 oz. |

The above arrangement is very convenient for accomplishing gold assays, and is the one employed in the assay office of the French Mint. The annexed cut (fig. 129) represents this apparatus.

The assay flask, *M*, being charged with the cornet, a constant amount of acid is added with a pipette. On the addition of the second acid a small piece of charcoal is placed in the flask: this serves to prevent bumping during ebullition. The flasks are supported on a plate of sheet

Fig. 129.



iron, *P*, pierced with holes, or by a grating, and the acid vapours, before escaping by the flue, pass into glass tubes, *T T*, about half an inch in diameter, and four feet long: at each end a narrower tube, *t*, is fused. The lower tube freely enters the neck of the flask; and as the space between is so small that a layer of acid remains suspended and obstructs the passage of the acid vapours, they are thus forced to pass into the large tube, where, for the greater part, they condense and fall into the flasks. By this means the quantity of acid employed in the assay can be diminished, as there is no loss by evaporation, and the results are found to be more constant. In order that the passage to the large tube for the acid vapours may always remain free, the end of the narrow tube passing into the flask must be cut at an angle (see *P*). The drops of acid collect at this part, and never close the tube.

For the assay of gold and silver alloys by Gay-Lussac's normal solution, see page 616.

*Assay of Tellurides and other native mineralised substances containing Gold.*—These assays are made in the scorifier in precisely the same manner as for silver substances of a like kind. The button resulting from cupellation is treated by quartation if necessary, and by nitric acid, as already described.

## BLOWPIPE REACTIONS OF GOLD.

The blowpipe reactions of gold are:—

**Graphic Gold.**—*On charcoal*, fuses into a dull grey metallic bead, covering the charcoal with a white smoke, which disappears with a green or bluish light, when the reducing flame is thrown upon it. After a continued blast, a bright yellow metallic grain is obtained. It is, after cooling, brilliant and malleable.

In the *open tube* it deposits a smoke, which is white, excepting in the neighbourhood of the assay, where it is greyish. This is sublimed tellurium. This deposit forms limpid drops when the flame is directed upon it.

**Telluriferous and Plumbiferous Gold.**—*Alone*, on charcoal it fuses like the preceding, and forms a pulverulent deposit on the support; but this deposit is yellow; it disappears in the reducing flame with a blue colour, which is not at all green. It gives, after a strong blast, a grain of gold, which ignites at the instant of congelation. This grain is malleable.

In the *tube* it fumes, giving a very sensible fume of sulphurous acid. It then gives a sublimate, which is grey close to the assay, but white elsewhere.

# CHAPTER XVII.

## THE ASSAY OF PLATINUM.

PLATINUM is found in a native or metallic state. It occurs very rarely, yet it is exceedingly probable that wherever gold is found this metal will more or less accompany it.

It is found disseminated in sand, in the form of grains varying in size from gunpowder to hempseed: this last size they rarely exceed; yet, as in the case of gold, nuggets have been found of large size and weight. Its colour is steel grey, or rather, a tinge between silver white and steel grey.

The sands from which Platinum is derived are remarkable, from the number and importance of their principal constituents. With the platinum may be found Au, Ag, Hg, Fe, Cu, Cr, Ti, Ir, Os, R, and Pd. Besides all these metals, precious stones have also been found associated with it.

*Analysis of Platinum Ores.*—The following is the method proposed by Berzelius. The operator first separates mechanically the particles of ore which differ in appearance. All those which are attractable by the magnet are next removed. Independently of the spangles of metallic iron which were first detected by Osann, the platinum sands often contain metallic compounds of iron and platinum, not only capable of being attracted by the magnet, but possessed even of polarity. These grains have a different composition from those not magnetic, as shown in the two following analyses by Berzelius.

Analysis of the non-magnetic grains :—

| | |
|---|---|
| Platinum | 78·94 |
| Iridium | 4·97 |
| Rhodium | ·86 |
| Palladium | ·28 |
| Iron | 11·04 |
| Copper | ·70 |
| Osmide of iridium in grains | 1·00 |
| in scales | ·96 |
| | 98·75 |

Analysis of the magnetic grains :—

| | |
|---|---|
| Platinum | 73·58 |
| Iridium | 2·35 |
| Rhodium | 1·15 |
| Palladium | ·30 |
| Iron | 12·98 |
| Copper | 5·20 |
| Insoluble matters | 2·30 |
| | 97·86 |

These grains being separated, their relative proportion is estimated.

The ore is to be treated with diluted hydrochloric acid.* The object of this is to free it from the coating of peroxide of iron with which it is often covered, and to dissolve the metallic iron. The quantity of iron separated from the ore in this manner is to be estimated.

The ore must not be ignited until it has previously been weighed; for during the ignition it generally acquires a coating of peroxide of iron, and a consequent increase of weight. It is sufficient to dry it upon a hot sand-bath.

The operator must not employ too large a quantity of the ore for analysis. Berzelius thinks about 30 grains is the best quantity. Sometimes, however, when the object is to determine with great accuracy the quantity of a constituent which occurs, but in a very small relative proportion, a larger quantity of the ore must be dissolved; but, in such a case, every other constituent is to be neglected.

Berzelius determines the solution of the weighed metal by

---

* At the mint in St. Petersburg they treat the ore with *aqua regia*; they use 10-15 parts of the latter, consisting of 3 parts muriatic acid of 1·18 spec. gravity and one part nitric acid of 1·04 spec. gravity, for one part of ore. The ore is digested in porcelain vessels for 8-10 hours, when it will be found to be dissolved.

*aqua regia*, in a glass retort furnished with a receiver, which is kept constantly cool. The acid distilled over during the solution is yellow, which colour does not proceed merely from the presence of chlorine, but from the constituents of the solution which are carried over mechanically.

The acid is distilled until the liquid has a syrupy consistence, and congeals on cooling. The saline mass so formed is dissolved in the smallest possible quantity of water, and the solution is poured off with all due precaution. The acid distilled over into the receiver is poured upon the undissolved portion of the ore in the retort, and again distilled. The second distillation generally effects the complete solution of the platiniferous matter.

If the distilled liquor be not colourless it must be returned into the retort and redistilled. The residue must be evaporated to a syrupy consistence as before, and treated with water. The distilled liquid generally contain a small portion of peroxide of osmium, of which a part is lost by the redistillation; its quantity, however, is in general very small.

The colourless distilled liquid is diluted with water, and saturated either with ammonia or with hydrate of lime. The acid, however, must remain a little in excess. The object of this saturation is to prevent the decomposition of the sulphuretted hydrogen gas, with which the solution is afterwards to be precipitated.

The precipitation is to be made in a flask which can be closely stoppered, and of such a size as to be nearly filled with the solution. When the solution contains free hydrosulphuric acid, the flask is closed, and left to itself until it is perfectly bright, which will be in about two days. The clear liquid is removed by a pipette, and the sulphide of osmium collected in a weighed filter, in which it is washed, dried, and weighed. According to theory, the resulting sulphide of osmium should contain 60·6 per cent. of that metal; but it is not obtained free either from moisture or excess of sulphur: it is also slightly oxidised during the process of drying. According to some experiments made by Berzelius with weighed quantities of this substance, it appears that the sulphide of osmium obtained by the operation just described contains from 50 to 52 per cent. of osmium. In general, however, the quantity of osmium is so small, that an error of a few hundredths in the reckoning of the quantity of osmium contained in this preparation is of no importance in regard to the analysis.

Respecting the metallic solution from the retort, it sometimes happens that after the saline mass has been dissolved in water, it smells slightly of chlorine.

This happens through the decomposition of a portion of the chloride of palladium. The solution must be allowed to digest until it no longer smells of chlorine. If the solution became troubled during the digestion, a portion of oxide of palladium is precipitated, which must be redissolved. The solution is filtered through a weighed filter, upon which is collected that portion which is undissolved. This portion consists of grains of osmide of iridium, of spangles of the same alloy, and of grains of sand, which could not be separated mechanically before the analysis. Sometimes, in addition to these, a black powder is found, which has the appearance of charcoal, and capable of passing through the filter during the washing of the other grains. This is peroxide of iridium, and is due to the presence of too much nitric acid in the *aqua regia*. As this occasions much extra work in the analysis, an excess of hydrochloric acid must be employed in making the *aqua regia*.

The filtered solution is now mixed with twice its bulk of alcohol, specific gravity ·833; so that the mixture may contain about 60 per cent. of its volume of alcohol. A very concentrated solution of chloride of potassium is now added, as long as it determines any precipitate.

The precipitate consists of the double chlorides of potassium and platinum, and of potassium and iridium, contaminated with rhodium and a little palladium.

The precipitate has a fine lemon-yellow colour when it is free from iridium; but when iridium is present it presents all shades, from deep yellow to cinnabar-red.

It is placed upon a filter, and washed with a mixture of alcohol (containing about 60 per cent. of anhydrous alcohol) and a small proportion of concentrated solution of chloride of potassium. The precipitate must be washed until the liquid passing through gives no precipitate with sulphuretted hydrogen.

The analysis is now divided into two distinct parts—the examination of the washed precipitate A, and treatment of the alcoholic liquid B.

A. The washed salt is dried, and carefully mixed with an equal weight of carbonate of soda. The filter, with that portion of the precipitate which it is impossible to separate from it, must be burnt, and the ashes mixed with a little carbonate of soda, and added to that mixed before. The whole is very gently heated in a porcelain crucible, until the mass is black throughout.

By acting thus the double salts are decomposed, and the platinum, whose oxygen passes away with the carbonic acid, is reduced. The rhodium and iridium meanwhile become oxidised, and remain in such a state as to permit of their separation from the platinum by solution.

When, instead of following the process just recommended, the precipitation of the double salts is effected by muriate of ammonia, the heating of the precipitate in a crucible not only reduces the platinum, but the rhodium and iridium also ; so that, on treating the heated mass with *aqua regia*, all three are dissolved.

The heated saline mass is washed with water until the greater mass of the saline contents is dissolved ; diluted hydrochloric acid is then added to the remainder to extract the alkali, combined with the oxides of iridium and rhodium. The mass is washed, dried, and ignited. The filter may be burnt, and an allowance made for the weight of the ashes ; but it is to be noted that the filter must be burnt by itself lest the metallic oxides be reduced. The mass is afterwards weighed.

When this is done the mass is mixed with five or six times its weight of bisulphate of potash, and fused in a platinum crucible. During the ignition the rhodium dissolves, and its solution is accompanied by an evolution of sulphurous acid gas. The platinum crucible must be kept closed during the ignition, by a cover which fits well, to check the too rapid volatilisation of the acid. As soon as the saline mass becomes fixed and crystalline at the surface when the cover is removed, the crucible must be taken from the fire and cooled. The salt is then dissolved in boiling water, and the undissolved residue is treated again with bisulphate of potash. The melted salt is red and transparent when it contains but little rhodium, but appears dark and black when it is nearly saturated with the metal. So long as the salt continues to become coloured, the re-melting must be repeated.

In order to avoid in analysis the employment of too large a quantity of bisulphate of potash, the operator may supply sulphuric acid as follows. When the bisulphate of potash appears to have lost the greater part of its free acid, weighed portions of the distilled sulphuric acid may be added to the mixture, the whole cautiously heated until the water of the acid be expelled, and the fusion thereupon be continued.

The quantity of rhodium can be determined by two methods. According to the first, the undissolved platinum is washed, ignited, and weighed, and the quantity dissolved is equal to the peroxide of rhodium, which contains 71 per cent. of metal; or the washings which contain the rhodium are super-saturated with carbonate of soda, evaporated to dryness, and ignited in a platinum capsule. If the mass be now acted on by water, peroxide of rhodium will remain. It may be collected in a filter, washed, dried, ignited, and finally reduced by means of hydrogen gas. The resulting metal is weighed. The rhodium thus obtained sometimes contains palladium. This is extracted by *aqua regia.* The solution of palladium is then neutralised and precipitated by cyanide of mercury: the precipitate is to be washed, dried, and ignited. The residual mass is metallic palladium, which may be weighed.

After the separation of the rhodium, the metallic mass is treated with very weak *aqua regia,* by digestion with which pure platinum is dissolved. The solution has a very deep colour, which is owing to the peroxide of iridium in supension: but when it has become bright by deposition it has a pure yellow colour. It is then decanted, and concentrated *aqua regia*, mixed with chloride of sodium, poured upon the residue. The solution is now evaporated to dryness. The addition of the chloride of sodium is to hinder the production of proto-chloride of platinum. A small quantity of iridium is dissolved in the very concentrated acid; but, if it were not used, a considerable portion of platinum would remain mixed with the iridium.

When the dry mass is acted on by water, peroxide of iridium remains unacted upon. If it were washed with pure water to dissolve out all the platinum, it would be carried through the pores of the filter; to prevent which a dilute solution of chloride of sodium must be employed; and to remove the least traces of that, solution of muriate of ammonia is used. The filter is now to be burnt, and the peroxide of iridium remaining, with its ashes, reduced to the metallic state by a current of hydrogen gas, and weighed.

The solution of chloride of sodium containing a small quantity of iridium is mixed with carbonate of soda, dried, and ignited. The product, freed from soda salts by water, and from platinum by weak *aqua regia*, leaves peroxide of iridium, which must be reduced to the metallic state, and added to that already obtained.

In order to arrive at the weight of platinum, the operator must deduct the weight of the peroxide of rhodium from the united weights of the peroxide of rhodium, peroxide of iridium, and platinum. He must then add to the weight of the iridium obtained 12 per cent. of the weight of that metal, to produce the weight of peroxide of iridium which must be deducted from the weight of the platinum.

The reduction of the platinum from its solution would only increase the length of the operation, without adding anything to its accuracy.

B. *Treatment of the Alcoholic Solution.*—This solution is poured into a flask capable of being well stoppered, and sulphuretted hydrogen passed in to saturation. It is then closed, and allowed to remain at rest for twelve hours in a warm place; at the end of which time all its metallic sulphides will be precipitated. Sometimes the solution is red, owing either to the presence of rhodium or sesquichloride of iridium.

The solution must now be filtered and evaporated to expel all alcohol, during which operation a little more metallic sulphide will be precipitated, and which must be added to that already obtained. The mixture of sulphides thus obtained, consists of the sulphides of iridium, rhodium, palladium, and copper; while the filtered solution contains iron, rhodium, iridium, and a trace of manganese. During the evaporation of the alcohol a greasy-like metallic sulphide of a disagreeable odour is deposited, which cannot be washed out. After the solution has been entirely washed away from this substance, it can be dissolved by pouring a little caustic ammonia into the capsule. The solution is now poured into a platinum crucible, and evaporated to dryness. The moist metallic sulphides are then placed in also, and roasted until all sulphurous acid is expelled. On the cessation of roasting, concentrated hydrochloric acid is poured over the mass; this, owing to the solution of subsulphate of copper and palladium, is coloured green or yellowish-green. Oxide of iridium and rhodium, with a little platinum, remain unacted upon.

The solution in hydrochloric acid is mixed with chloride of potassium and nitric acid, and evaporated nearly to dryness; a dark saline mass is the result, which is composed of chloride of potassium and cupreo-chloride of potassium, with palladio-chloride of potassium. The two first of these salts are dissolved out in alcohol, specific gravity ·833, and the palladium salt is placed on a filter and washed with alcohol of the same specific gravity. It contains 28·84 per cent. of palladium when dried and ignited.

The spirituous solution, which contains the copper salt, is evaporated to get rid of alcohol; and the contained copper is precipitated, either by means of pure potash, or by adding sulphuric acid and a plate of zinc.

That portion of the roasted sulphides which was insoluble in hydrochloric acid is fused with bisulphate of potash until it ceases to become coloured. The mixture, in this case, contains much more rhodium than the precipitate obtained at the commencement of the analysis. The residue undissolved by bisulphate of potash, which is peroxide of iridium with a little platinum, is treated with *aqua regia*, and the peroxide reduced by hydrogen gas, as stated in a former part of the analysis.

The concentrated solution from which the sulphides were precipitated contains only iron in a state of protochloride, with a small quantity of iridium and rhodium, and a trace of manganese. It must be mixed with a proper quantity of nitric acid, and boiled till the iron is fully oxidised. The peroxide of iron is then precipitated by caustic ammonia, and the precipitate washed, ignited, and weighed.

This peroxide of iron, however, contains a small quantity of iridium and rhodium, to separate which, after weighing the peroxide, it must be reduced by hydrogen gas. The reduced metal is treated with hydrochloric acid to dissolve iron, and the black undissolved portion is collected on a filter, ignited with exposure to air, and weighed; its weight deducted from that of the peroxide of iron, previously obtained, leaves the quantity of the latter in a pure state. The solution, filtered from the precipitate by ammonia, is mixed with carbonate of soda in sufficient quantity to decompose the ammoniacal salts, and evaporated to dryness. On treating the residue with water, after a gentle ignition, peroxides of iridium and rhodium remain undissolved; but they are generally too small for separation.

The following plan will serve to detect platinum in admixture with gold and other heavy matters obtained by washing or vanning sands, earths, &c.:—

Act on a small quantity by mercury, and separate the amalgam: by this means the gold is removed. To the residue add *aqua regia*, and boil; evaporate the solution to dryness; add a little muriatic acid and water; boil and filter. To the filtered solution add a strong solution of sal ammoniac (chloride of ammonium). If a bright yellow, or reddish-yellow, granular precipitate falls, platinum is present in the sand.

A still more ready method is the following: Separate as much earthy matter as possible by careful washing. If gold is present, separate that by amalgamation. Dry the residue, and take its specific gravity: if it be above 10, platinum is most likely present. The specific gravity of native platinum, free from earthy matter, is from 16 to 19.

The following method of Analysis of Platinum Ores, by MM. Deville and H. Debray, is of interest. The ores of platinum contain the following substances:—

1. Sand. The whole of the sand is never removed by washing the ore; and the sand contains quartz, zirconium, chromate of iron, and, in the Russian ores, titanate of iron.

2. Osmide of iridium.

3. Platinum, iridium, rhodium, and palladium, combined, no doubt, in the form of an alloy.

4. Copper and iron, which exist in the ores in a metallic state, for the iron found in the sand is not soluble in acids.

5. Gold, and, oftener than is supposed, a little silver. The latter metal is generally found with the palladium, and it is very rarely that palladium is obtained quite free from silver when it is prepared by the old processes.

1. *Sand.*—To estimate the sand we take a small assay crucible, or an ordinary crucible with smooth sides, and melt in it a little borax, so as to glaze the inside. We now introduce from 7 to 10 grammes of pure granulated silver, and 2 grammes of the ore fairly taken and weighed very accurately. Over the platinum we put 10 grammes of fused borax, and one or two small pieces of wood charcoal. The silver is now melted, and care must be taken to keep it for some time a little hotter than the melting point, so that the borax may be very liquid, and may dissolve the vitreous matters which accompany the platinum and constitute the sand. The crucible is now allowed to cool, and when it is cold, the button, which will contain the silver, osmium, platinum, and all the other metals, is detached, and if necessary digested for a time with weak fluoric acid to remove the last portions of borax. It is now heated to a faint redness, and then weighed. The weight of the button, subtracted from the sum of the weight of the ore and silver employed, will give the amount of sand contained in the ore. For example:—

| | Milligr. |
|---|---|
| Californian ore . . . . . . . | 2000 |
| Silver . . . . . . . . . | 7221 |
| | 9221 |
| After fusion, the button weighed . . | 9162 |
| Consequently, the ore contained, sand . . | 59 |

It is very important to know this number, for it represents the only matter absolutely destitute of valüe which the ore contains; and this simple operation may be considered the most important performed in estimating the value of an ore. It is, besides, performed so quickly that it is as well to do at the same time two or three specimens, taken from different parts of a lot of platinum powder.

2. *Osmide of Iridium.*—Another 2 grammes of the ore weighed very accurately are treated with *aqua regia* at 70° (Cent.) until the platinum is entirely dissolved. The *aqua regia* must be renewed occasionally for 12 or 15 hours, or until it is no longer coloured. It is best to perform this operation in a large beaker, and to place a cover over it to prevent loss. The solution must be decanted with the greatest care from the metallic spangles of the osmide of iridium and the sand which remain at the bottom of the beaker. If necessary it may be filtered, but as little as possible of the osmide must be allowed to go on the paper. The insoluble residue must be washed by decantation, then dried and weighed, after having added what remained on the filter. By subtracting the weight of the residue from the weight of the sand obtained in the former operation, we obtain the weight of the osmide of iridium. For instance, in the Californian ore we had:—

| | Milligr. |
|---|---|
| Osmide of iridium and sand . . . . | 81 |
| Sand . . . . . . . . . . | 59 |
| Osmide of iridium . . . . . . . | 22 |

The button obtained in determining the sand might be employed in this operation. In that case it is necessary to dissolve out the silver with nitric acid, and then proceed with the residue, as we have just directed.

3. *Platinum and Iridium.*—The solution in *aqua regia* obtained in the last operation is evaporated to dryness at a

low temperature, and the residue is redissolved in a small quantity of water (if it should not entirely dissolve in the water, some more *aqua regia* must be added, and the evaporation repeated), to which is added about twice as much pure alcohol; lastly, we add a great excess of sal ammoniac in crystals. The whole is now slightly warmed to complete the solution of the sal ammoniac, it is then stirred, and afterwards set aside for 24 hours. The orange-yellow, or even reddish-brown precipitate, which is formed contains the platinum and the iridium, but some remains in the solution. The precipitate must be thrown on a filter and washed with alcohol. Afterwards the filter is dried in a platinum crucible, placed, for greater safety, within a larger one, and afterwards heated by degrees to low redness. The crucibles are now uncovered, and the filter is burnt at the lowest possible temperature. Once or twice after the incineration of the filter a piece of paper saturated with turpentine should be introduced into the crucible, by which means the oxide of iridium will be reduced, and the expulsion of the last traces of osmium will be effected. The crucible is now heated to whiteness until it no longer loses weight, or the reduction is finished in a current of hydrogen.

The liquid separated from the platinum-yellow by filtration, is evaporated until the chloride of ammonium crystallises in great quantity. It is allowed to cool, is then decanted, and on a filter is collected a small quantity of a deep violet-coloured salt, which is the ammonio-chloride of iridium mixed with a little of the platinum salt. This is first washed with a solution of sal ammoniac, and then with alcohol. The salt is then ignited, and if necessary reduced by hydrogen like the platinum salt. The mixture of platinum and iridium obtained by the two reductions is then weighed. The two metals are now digested at about 40° or 50° (Cent.) in *aqua regia*, diluted with about 4 or 5 times its weight of water—the *aqua regia* being renewed until it is no longer coloured. The residue is pure iridium. To obtain the weight of the platinum the weight of the iridium is subtracted from that of the mixture of the two. This method of separating the two metals is very accurate if the *aqua regia* used be weak, and the contact with it prolonged.

4. *Palladium, Iron and Copper.*—The liquid charged with sal ammoniac and alcohol, from which the platinum and iridium have been separated, is evaporated to get rid of the alcohol, and then treated with an excess of nitric acid, which transforms the chloride of ammonium into nitrogen and hydrochloric acid. It is now evaporated almost to dryness. The residue is removed to a covered porcelain crucible which is weighed with great care. When the matter is dry it is moistened with concentrated hydrosulphuret of ammonia and afterwards dusted over with 2 or 3 grammes of pure sulphur. When dry, this crucible is placed within a larger one of clay, and surrounded with pieces of wood charcoal. The two, covered, are now set in a cold furnace which is filled up with charcoal, and the fire is lighted at the top to avoid the projection of any matter from the crucible, if it were too quickly heated. After reaching a bright red heat, the crucibles are allowed to cool. The porcelain crucible now contains palladium in a metallic state, with the sulphides of iron and copper, and also the gold and rhodium. This mixture is moistened with concentrated nitric acid, which, after prolonged digestion at 70°, dissolves the palladium, iron, and copper, forming at the same time a little sulphuric acid. The solution of the nitrates is poured off the residue which is washed by decantation, and the solutions and washings are evaporated to dryness, and then calcined at a strong red heat. In this way the palladium is reduced, and the iron and copper pass to the state of oxides, which are easily separated from the palladium by means of strong hydrochloric acid. The palladium remains in the crucible in which it is again strongly ignited and then weighed.

The chlorides of iron and copper are now evaporated to dryness at a temperature but little above 100° (Cent.) and are then treated with ammonia. The sesquichloride of iron having lost nearly all its acid, has become insoluble; but the chloride of copper is readily dissolved, and may be filtered from the iron which is washed, ignited, and weighed.

The copper solution is now evaporated almost to dryness, and then mixed with excess of nitric acid, and heated to drive off the chloride of ammonium. Afterwards the nitrate of copper is ignited and weighed. The weight of the copper is always so small that the hygrometric water the oxide of copper may absorb may be neglected.

5. *Gold and Platinum.*—The residue insoluble in nitric acid is weighed and treated with very dilute *aqua regia.* which takes up the gold, and sometimes, but very rarely, traces of platinum. To ascertain if platinum be present, evaporate to dryness, and re-dissolve by alcohol and chloride of ammonium. If any platinum-yellow remain, it must be ignited and weighed. The difference in the weight of the porcelain crucible before and after the treatment by *aqua regia*, gives the weight of the gold, from which, if any be found, the weight of the platinum must be deducted.

6. *Rhodium.*—The residue left in the crucible is rhodium, which must be reduced in a current of hydrogen.

We append the results of some analyses of platinum ores, by MM. Deville and Debray.

ANALYSES OF PLATINUM ORES FROM VARIOUS SOURCES.

| | Columbia | California | Oregon | Spain | Australia | Russia |
|---|---|---|---|---|---|---|
| Platinum . . | 80·00 | 79·85 | 51·45 | 45·70 | 59·80 | 77·50 |
| Iridium . . | 1·55 | 4·20 | 0·40 | 0·95 | 2·20 | 1·45 |
| Rhodium . . | 2·50 | 0·65 | 0·65 | 2·65 | 1·50 | 2·80 |
| Palladium . . | 1·00 | 1·95 | 0·15 | 0·85 | 1·50 | 0·85 |
| Gold . . . | 1·50 | 0·55 | 0·85 | 3·15 | 2·40 | (*) |
| Copper . . | 0·65 | 0·75 | 2·15 | 1·05 | 1·10 | 2·15 |
| Iron . . . | 7·20 | 4·95 | 4·30 | 6·80 | 4·30 | 9·60 |
| Osmide of iridium | 1·40 | 4·95 | 37·30 | 2·85 | 25·00 | 2·35 |
| Sand . . . | 4·35 | 2·60 | 3·00 | 35·95 | 1·20 | 1·00 |
| Osmium and loss . | . . | 0·05 | . . | 0·05 | 0·80 | 2·30 |
| | 100·15 | 100·00 | 100·25 | 100·00 | 100·00 | 100·00 |

M. A. Guyard gives the following process for the extraction of metals from platiniferous residues.

'This process comprises three different operations, which I will succinctly describe.

'1. *Solution of the Residues.*—The mother liquors which remain after the precipitation of platinum by sal ammoniac

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[*] Gold, if any, counted in the loss.

come from solutions of crude or commercial platinum. They always contain iron, mostly produced from the sulphate of iron used for the precipitation of gold, lead, copper, palladium; platinum, and especially rhodium. These mother liquors are acidulated by hydrochloric acid, and are then ready to be investigated. To recall their composition, I shall distinguish them here only as residues in solution. It need only be mentioned that iron, which is generally used for the precipitation, must be avoided.

'Solid residues are melted at once with three times their weight of a mixture of equal parts of soda and nitrate of soda. The fusion is effected at a bright red heat in a thick iron vessel. It is accomplished without bubbling or projection, and requires about an hour. During the last twenty minutes the mass must be constantly stirred with an iron spoon. The operation is extremely simple.

'These residues contain osmide of iridium, unattackable by all chemical agents, attackable osmide, some grains of triple alloy of platinum, iridium, and rhodium, which *aqua regia* will not dissolve, but which nitre completely oxidises and completely breaks up. They also contain the gangue characteristic of platinum ores—quartz, silicates of all bases, titanates, hyacinths, &c. &c.

'The mixture I make use of oxidises all that is oxidisable, and breaks up the gangue, which it partly dissolves. The melted mass contains all the bodies above mentioned, besides a large quantity of oxide of iron, taken from the sides of the vessel in which the operation is performed. The fused mass is poured into cast-iron moulds. When solid it is broken into fragments and boiled with sufficient water to obtain a strong solution of soda, capable of holding all the gelatinous acids in solution. It also contains osmium in the state of osmiate.* It is filtered from insoluble matter, and then supersaturated with hydrochloric acid. The insoluble oxides are freed by washing from the excess of alkali and are then dissolved in *aqua regia*.

'This solution contains iron, copper, lead, iridium, rhodium, platinum, and ruthenium. It is separated from the undissolved osmide, evaporated to expel the excess of *aqua regia*, and dissolved in water and hydrochloric acid.

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* This solution is separately precipitated by hydrosulphuric acid. Sulphide of osmium is thus isolated.

'2. *Precipitation of Liquids by Sulphuretted Hydrogen.*—Liquids obtained as above are ready for precipitation by hydrosulphuric acid.

'The apparatus in which all the liquids are precipitated is composed of a sulphuretted hydrogen gas generator by the action of sulphuric acid on sulphide of iron. This generator communicates with four or five large earthenware jars, holding about 70 litres, arranged precisely as in Wolff's apparatus. A special tube conducts to each of them the vapour destined to heat the liquid which they contain.

'The whole apparatus is enclosed in a well-fitted wood stove placed near a chimney, with which it communicates. As to the small quantities of unabsorbed gas, they are conducted into the chimney, where the fire creates a strong draught. By this means, also, all smell is avoided during the precipitation; but after the operation air is forced through the apparatus from large gasometers. It expels the hydrochloric acid which saturates the mother waters, and these can then be manipulated free from smell.*

'The experiment is carried on during the precipitation in the following manner: when the generator begins to disengage gas, the temperature of the liquids is raised to about 70°. This temperature is maintained for nearly fifteen hours, that being the time required for the complete precipitation of the sulphides, which collect better under the influence of heat. The operation is concluded when there remains but a very slight yellow tint in the mother liquor, arising from the presence of a little soluble sulphide of iridium. This mother liquor is poured from the precipitated sulphides into a vessel with pieces of iron, which takes off a little of the iridium. The sulphides are filtered through linen filters.

'3. *Purification and Treatment of the Sulphides.*—The mass of sulphides thus separated from the iron and from all other bodies not precipitated by the sulphuretted gas, contains, in addition to the sulphides of the platinum metals, a large proportion of sulphur and the sulphides of copper and lead. To get rid of these bodies, I have thought of concentrated sulphuric acid, which changes them to sulphurous acid and sulphates, while it does not act on the sulphides of the precious metals. This refining can be effected in an iron vessel, but Mr. Matthey, who neglects nothing to ensure the certainty and exactness of the results, makes use of platinum.

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* A carbonic acid generator may be substituted for the gasometers and the air with no difference in the result.

'When, after prolonged boiling, no more sulphurous acid is given off, the refining is complete.

'The mass of sulphides, diluted with a quantity of water, is thrown on filters, and thoroughly washed, until ammonia no longer finds any trace of copper or iron in the filtered liquid.

'At this point precious metals are entirely freed from iron, which is so detrimental to them, and from copper, and contain only a little sulphate of lead, which separates by itself during an ulterior reaction. They are then, moreover, in a condition to be dissolved by simple nitric acid or by *aqua regia*, and this is not their least valuable condition.

'*Treatment of the Sulphides.*—The sulphides are next dissolved in *aqua regia*, which should not be previously prepared, because its action on sulphates is so sudden and energetic; it heats so rapidly, and the disengagement of gas is so great that, were it previously prepared, it would certainly be thrown from the vessels.

'I add then moderately strong cold nitric acid, and add it gradually, because its action is strong. A quantity of rutilant vapours are disengaged. Hydrochloric acid is added when the effervescence ceases. It is then gradually heated to boiling, which is necessary to obtain a complete solution.

'The solution is poured from the deposited chloride of lead, and the ordinary method with sal ammoniac is used to separate the different metals it contains. Experiments on large quantities of material have fully proved the advantages of this process.'

## CHAPTER XVIII.

### THE ASSAY OF BISMUTH.

THE FOLLOWING varieties of bismuth ores are met with, but are somewhat rare :—

Oxide of Bismuth.
Sulphide of Bismuth.
Persulphide of Bismuth.
Cupriferous Sulphide of Bismuth.
Plumbo-cupriferous Sulphide of Bismuth.
Plumbo-argentiferous Sulphide of Bismuth.

Lastly, we have Native Bismuth, which, although far from common, is the only mineral hitherto found to supply the wants of commerce with the pure metal; and the only products of it are bismuth slags and cupel bottoms, in which oxide of bismuth is present in lieu of oxide of lead ; it sometimes happening that bismuth is employed instead of lead in cupellation (see Silver Assay).

*Native Bismuth* possesses a tolerably bright metallic lustre; its colour yellowish-white, often iridescent. It fuses in the candle flame. It is generally found in small amorphous lamellar masses, yet it occasionally occurs in acute rhomboidal as well as cubical and octohedral crystals.

This substance does not seem to form veins by itself, but generally accompanies other minerals, particularly those of cobalt, nickel, arsenic, and lead.

*Assay of Native Bismuth.*—The assay of native bismuth may be done in the same way as that of Antimonium crudum, i.e. the bismuth is separated from the gangue in which it occurs by heating the mineral in closed vessels, as described at the Assay of Antimony.

*Assay of Bismuth Residues, Cupel Bottoms, &c.*—These

substances must be finely pulverised, and from 200 to 400 grains mixed with three times its weight of fused borax, their own weight of carbonate of soda, and from 100 to 200 grains of cyanide of potassium, and proceed with all the precautions above pointed out.

In case the mineral contained besides bismuth other metals (they are mostly tin, copper, and lead), the resulting metal-button will contain part of those metals, and they must be determined by the humid process.

*Determination of amount of Bismuth by the Humid Process.*—Act on 50 grains of the finely powdered substance with strong nitric acid until all action ceases, evaporate to dryness, add from 50 to 100 drops of strong sulphuric acid, well mix with a glass rod, and evaporate to dryness; add water, with a few drops of sulphuric acid, and boil. Filter the solution, and to the filtered solution add excess of carbonate of ammonia. Collect the oxide of bismuth thus thrown down on a filter, wash, and dry; separate it carefully from the filter, ignite it, and weigh: every 100 parts correspond to 89·87 of bismuth.

Or the bismuth may be obtained at once in the metallic state from the solution prepared as above: by adding to it metallic copper in the form of a small sheet, and gently heating, the bismuth will separate in the metallic state, and can be washed, dried, and weighed, as directed for copper, under the Assay of that metal.

The high price of bismuth for some years past has induced M. Balard to undertake the search for this metal in old type metal. When it was cheaper, bismuth entered into the composition of the alloy for printing purposes. M. Balard proposes to effect this industrial analysis in the following way:—

1. Dissolve the material in nitric acid, so as to transform all the tin into metastannic acid, which isolate by filtration from the acid solution of nitrates of lead and bismuth; wash with acidulated water, dry, and reduce by charcoal.

2. Into the liquid, neutralised as much as possible, plunge plates of lead, which precipitate all the bismuth in a metallic state; dry, and melt with a reducing agent.

3. Precipitate the lead from the last liquid by carbonate of soda; separate, wash, dry, and reduce with charcoal.

This way of operating gives the three metals in a metallic state; it may undergo several modifications for isolating the metals under another form according to the arrangement of the products. To obtain extremely pure subnitrate of bismuth, says M. Balard, it is necessary only to neutralise the liquid containing the soluble nitrates, and dilute with a large quantity of water naturally free from carbonates, chlorides, or sulphates. After again neutralising and diluting with water and repeating the operations several times, the greater part of this metal becomes separated in the state of white bismuth.

Mr. R. W. Pearson has given the following process for the Assay of Bismuth by weight and by volume.

*Preparation of standard solution.*—·7135 grs. of pure crystallised bichromate of potash are dissolved in 100 grs. of water. Call this solution the bichrome test A.

In a similar way, prepare a second solution, one-tenth the strength of bichrome test A; ·07135 grs. of bichromate of potash diffused in 100 grs. of water, will furnish such a solution; call it the bichrome test B. Bichrome test C, one-tenth the strength of solution B, is also prepared by dissolving ·007135 grs. of the bichromate of potash in 100 grs. of water.

These figures can be multiplied to any convenient number. These solutions will contain bichromate of potash, in 100 grs. of bichrome test A, equal to 1 gr. of bismuth; in 100 grs. of bichrome test B, equal to 0·1 gr. of bismuth; and in 100 grs. of bichrome test C, equal to ·01 gr. of bismuth.

The bismuth should be in the form of nitrate, and the solution kept hot during the experiment, as the precipitated chromate collects more readily than after comple precipitation of the bismuth; the solution will exhibit a characteristic colour, produced by excess of the bichromate of potash.

By employing a standard solution of bismuth it has been ascertained that 71·35 parts of bichromate of potash are required to combine with 100 parts of bismuth.

## BLOWPIPE REACTIONS OF BISMUTH.

**Native Bismuth.**—*Alone*, fuses, giving a weak arsenical odour. Otherwise, it presents the same phenomena as pure bismuth.

In the open tube it gives a little arsenious acid. Cupelled, it tinges the bone ash pure orange-yellow.

**Sulphide of Bismuth.**—*Alone*, in the tube, gives sulphurous acid and a white sublimate; heated to redness, it deposits oxide of bismuth round the assay, like pure bismuth. On charcoal it fuses with bubbling, throwing out small incandescent globules. This agitation lasts but a short time.

**Oxide of Bismuth.**—*Alone*, oxide of bismuth fuses readily on the platinum wire, forming a deep brown mass, which becomes yellow on cooling. If acted upon by a very intense flame, it is reduced, and perforates the platinum. It is reduced instantaneously on charcoal.

*With borax* it fuses into a colourless glass in the oxidising flame. In the reducing flame it becomes greyish, owing to the dissemination of particles of bismuth.

*Microcosmic salt* forms with it a brownish-yellow glass. In the reducing flame, particularly with tin, a glass is formed, which is clear and colourless while hot, but becomes greyish-black on cooling. Oxide of copper presents nearly the same phenomena under the same circumstances, but with this difference—that tin produces a red colour.

Owing to the facility with which bismuth may be reduced, it is nearly always on the metal that the assay is made; hence it becomes very important to distinguish it from the antimony and tellurium, with which it may be readily confounded.

Firstly, in the matrass neither antimony nor bismuth sublime at a temperature the glass can bear. Tellurium, on the contrary, gives at once a little smoke (by means of the oxygen of the atmosphere), and finally, a grey sublimate of metallic tellurium is obtained.

Secondly, in the open tube antimony gives a white vapour, which lines the interior of the tube, and which can be driven by heat from one part to another without leaving the least trace. The metallic bead is always covered by a notable quantity of fused oxide.

Tellurium gives much vapour, which attaches itself to the sides of the tube as a white powder, which is capable of fusion into colourless drops by the application of heat.

Bismuth gives no smoke if it be not combined with sulphur; and the fused metal is surrounded by the brown fused oxide, which strongly attacks the glass.

Thirdly, on charcoal these three metals give off vapour by the action of heat, and leave a ring around the spot on which they are placed. That from antimony is quite white; those from bismuth and tellurium, red or orange. If the reducing flame be made to play upon them they disappear, at the same time colouring the flame a deep green if tellurium be present, and pale bluish-green if antimony. It is not coloured at all by bismuth.

# CHAPTER XIX.

## THE ASSAY OF CHROMIUM.

**The principal** ore of this metal which occurs in commerce is known as chrome iron, or chrome iron ore. It is found in amorphous masses of a brownish-black colour, approaching an iron grey. Its fracture is uneven, sometimes lamellar; and its powder is greyish.

The two following analyses will give a general idea of its composition :—

| | | |
|---|---|---|
| Oxide of chromium . . . . | 36·0 | 43·7 |
| Peroxide of iron . . . . | 37·0 | 34·7 |
| Alumina . . . . . . | 21·5 | 20·3 |
| Silica . . . . . . . | 5·0 | 2·0 |
| | 99·5 | 100·7 |

### *Assay of Chrome Ore.*

Chrome iron ore, like native oxide of tin, is very difficultly decomposable by ordinary re-agents. A good method of operating is thus:—Mix 50 grains of ore, reduced to the utmost state of division, with 100 grains of nitrate of potash and 200 grains of carbonate of soda; place the mixture in a platinum crucible, and expose to a red heat for half an hour; remove the crucible, and allow it to cool. Place it, when cold, in an evaporating basin, and add enough water to cover the crucible: gradually heat the basin and contents to ebullition. The fused mass in the crucible will gradually dissolve, and if the operation has been successful there will be no undecomposed chrome ore: if, however, there be, it must be collected, as in the case of the analysis of tin ore, dried again, ignited with nitrate of potash and carbonate of soda, and treated with water, as just described. The solution which is obtained is deep yellow, its colour being due to chromate of potash and soda, which have been formed at the expense of the oxygen of the nitric acid, which has converted the oxide of chromium into chromic acid: thus—

$$Cr_2O_3 + 3O = CrO_3;$$

and the chromic acid so produced combines with potash and soda to form the chromates, having the following formula :—

$$KO,CrO_3 + NaO,CrO_3.$$

The solution is to be filtered from the insoluble residue, consisting principally of peroxide of iron, and evaporated to dryness with small excess of nitric acid: the dry mass is treated with water, and the whole boiled, and, if necessary, filtered. It must now be treated with solution of proto-nitrate of mercury, which throws down chromate of mercury: the proto-nitrate must be added as long as a precipitate is produced. The chromate of mercury is collected on a filter, well washed, dried, and ignited. During the process of ignition the chromate of mercury is decomposed into mercury and oxide of chromium of a pure bright green colour. 100 parts of this oxide correspond to 70 parts of metallic chromium.

Calvert used for decomposition of chrome iron ore a mixture of 3-4 parts of caustic soda, and 1 part soda-nitre. He heated the ore with such mixture for 2 hours in a platinum crucible.

The following process, as described by F. W. Clarke, S.B., has given excellent results.

One part of the finely pulverised mineral is mixed in a platinum crucible with three parts of fluoride of sodium, and upon the top of this mixture are placed twelve parts of bisulphate of potash, which may be either in powder or in small lumps.

Upon heating, the mixture boils up strongly, and after a while settles into a clear, tranquil fusion. The boiling is chiefly owing to the action of the reagents upon the mineral, and not, as might be supposed, merely to the influence of the bisulphate upon the fluoride. This is shown by the fact that, whenever the reagents are heated together without minerals, although some boiling takes place, the addition of a little powdered chrome iron ore fully doubles the violence of the action.

In quantitative analyses it is necessary to keep the crucible closely covered, in order to avoid loss from spattering; and to heat carefully, so that the mass may not boil over. The bisulphate should never be mixed with the fluoride and mineral, because a portion of the assay is then apt to escape action, being left on the sides of the crucible by the boiling of the mass; but should be placed upon the top of the mixture as above directed, as then the decomposition is complete. The mass obtained by this fusion is, in the case of some minerals, completely soluble in water. In other cases, basic salts are formed, which, although insoluble in water, dissolve readily in hydrochloric acid. Almost all of the latter class may be rendered soluble in water by the following process: The fused mass, after cooling, without removal from the crucible, is treated with a small quantity of strong sulphuric acid, and again fused. The mass thus obtained is entirely soluble in water. There are exceptions to this rule, however.

The following results have been obtained. For the sake of brevity, we will speak of the fusion with bisulphate and fluoride as fusion No. 1, and the subsequent treatment with sulphuric acid, as fusion No. 2.

*Fusion No.* 1.—Chrome iron ore is decomposed very easily. In one case, in which the operation was timed, the fusion was complete in less than three minutes from the time the heating was commenced, and that over an ordinary Bunsen's gas burner. The cooled mass is light green, partly soluble in water alone, and entirely soluble in hydrochloric acid.

*Fusion No.* 2.—The mass possesses a deeper green colour than that obtained by the first fusion, and a larger proportion of it dissolves in water. In every fusion that I have yet made of chromite, however, a small quantity of basic salts was formed, requiring treatment with hydrochloric acid.

For the technical determination of chromium in chromite, Mr. Clarke says: After fusion with cryolite and bisulphate of potash, as previously directed, the mass is to be treated with a little strong hydrochloric acid, and allowed to digest for about ten minutes. Then upon boiling with water, the whole dissolves. The solution should then be neutralised, acetate of soda added, and the chromium oxidised to chromic acid by a current of chlorine gas, or by boiling with hypochlorite of soda solution. The chromium may then be separated from other substances, as directed in Professor Gibbs's paper ('Am. Journ. Sci.,' January 1865). When chromite is fused with bisulphate of potash and cryolite, and saltpetre is added to the mass, as soon as clear fusion is obtained, the chromium is nearly all oxidised to chromic acid. If the mass be boiled with a solution of carbonate of soda, and the liquid filtered, a filtrate is obtained which contains nearly all, but not quite all, the chromium as alkaline chromates, free from iron or alumina; but, invariably, the residue upon the filter contains traces of chromium. When chromite is fused with the acid fluoride of potassium, a part of the chromium is usually oxidised to chromic acid by the oxygen of the air; and in one case that came under my observation, when I came to heat the resulting mass with sulphuric acid, red fumes were given off, which were probably the so-called terfluoride of chromium.

When bisulphate of potash *alone* is used for the decomposition of chromite, &c., it is necessary that the mineral should be reduced to extremely fine powder; but when the mixture of bisulphate and fluoride is employed, although the mineral should be in fine powder, such an extreme state of subdivision is by no means required, and thus much labour is saved.

*Determination of Chromium by means of Standard Solution.*—This process is the converse of the determination of iron by means of solution of chromate of potash.

The chrome ore is treated with nitrate of potash and carbonate of soda, as above described; and the solution of chromate of potash so obtained has an excess of hydrochloric acid added to it.

It is stated at page 276, under the head of Iron Assay by Standard Solution, that 100 parts of metallic iron correspond to and are represented by 88·6 grains of bichromate of potash: now 88·6 grains of bichromate of potash contain 32·96 grains of chromium; therefore 100 grains of iron are equal to 32·96 of chromium. From these data a standard solution can be readily made: thus—Dissolve 50 grains of harpsichord wire in excess of hydrochloric acid; place the solution in the burette, and fill up to 100 on the instrument with water, and well mix: it is now evident that every division of the burette will equal or represent ·1648 grains of chromium. The assay is now thus proceeded with: Gradually add the standard solution of iron to the solution of chromate of potash (or rather, now, bichromate of potash) acidulated with hydrochloric acid, until a drop of the solution mixed with a drop of solution of ferrocyanide of potassium gives a pale blue colour: a slight excess of protoxide of iron is then present, showing that all the chromic acid has been reduced to the state of oxide of chromium. Now observe how many divisions of the iron solution have been required, and multiply them by ·1648: the resulting number will represent the amount of metallic chromium in the sample submitted to assay.

## BLOWPIPE REACTIONS OF CHROMIUM.

### ORES OF CHROMIUM.

**CHROME OCHRE.**—*Alone*, decolourises, and becomes nearly white, but does not fuse.

*Borax* separates oxide of chromium, and takes a fine green colour.

It dissolves with great difficulty in microcosmic salt, and the green colour is not so beautiful as with borax.

**OXIDE OF CHROMIUM.**—*Alone*, undergoes no change.

*With borax*, fuses difficultly, even in small quantity. The glass has a splendid emerald-green colour, which is principally developed during cooling.

*With microcosmic salt* it fuses in the exterior as well as in the interior flame, furnishing a deep green glass; and a very small quantity of oxide suffices to produce this effect.

*Soda* dissolves oxide of chromium on the platinum wire in the exterior flame, producing a deep orange glass, which becomes yellow on cooling. In the reducing flame it becomes opaque. It is green after cooling.

# CHAPTER XX.

## THE ASSAY OF ARSENIC.

THE MINERALS from which arsenic is produced are the following :—

Native arsenic.
Arsenic kies, $FeS^2 + FeAs$, containing 46,6 As and 19,6 S.
Arsenical kies, $Fe_4As_3$, containing 66,8 As.
Speiskobalt (Co,Ni,Fe), As.
Glanzkobalt, $CoS_2 + CoAs$.
Coppernickel, $Ni_2As$.
Nickel and cobalt arsen-kies, $(Co,Ni,Fe)S_2 + (Co,Ni,Fe)As$.
White nickel-kies, NiAs; Tennantite $(Cu_2S,SeS)^4,AsS_3$.
Realgar $AsS_2$ and yellow arsenic $AsS_3$.

*Assay for Arsenic.*—50 grains of the finely pulverised mineral are deflagrated with 200 of nitrate of potash and 200 of carbonate of soda in a porcelain crucible. When the crucible is cold, it and its contents are to be treated with water, as in the case of chromium. The solution will contain arseniate, and (if the ore had in its constitution sulphur, which is most likely) sulphate of potash. Nitrate of lead must be added to the solution (made neutral with nitric acid, if requisite): a mixture of arseniate and sulphate of lead is precipitated: this precipitate is well washed on a filter, and digested with dilute nitric acid: this agent dissolves out the arseniate of lead, and leaves the sulphate. Filter, and saturate the filtered solution with soda, which will throw down the arseniate: this must be collected on a filter, washed, dried, and weighed. Every 100 parts correspond to 22·2 of metallic arsenic, or 29 parts of arsenious acid (the common white arsenic of the shops).

This method is only approximative: the following is the better plan :—

Digest the ore in strong nitric acid until nothing more is taken up (the action may be facilitated by the occasional addition of a crystal or two of chlorate of potash), and all action on the addition of fresh acid is at an end : dilute with water, and filter : to the filtered solution add nitrate of lead, and proceed as above.

# CHAPTER XXI.

## THE ASSAY OF MANGANESE.

THE FOLLOWING are the commercially valuable minerals containing manganese.

| | | | | | |
|---|---|---|---|---|---|
| Pyrolusite, | $MnO_2$, | containing | 18 | p.c. of available | oxygen. |
| Braunite, | $Mn_2O_3$ | ” | 10·0 | ” | ” |
| Manganite, | $Mn_2O_3$, | ” | 9 | ” | ” |
| Varvicite, | $MnO_2+Mn\ O_3,HO$, | ” | 13·8 | ” | ” |
| Hausmannite, | $MnO+Mn_2O_3$, | ” | 6·8 | ” | ” |
| Psilomelane, | $Mn_2O_3$. | | | | |

*Assay of Manganese Ores.*—The assay of this metal is confined to the amount of peroxide any one of its ores may contain. There are several methods of affecting this, and the best of these will be described below.

The following method is described in Graham's 'Elements of Chemistry,' page 536 :—

The value of the oxides of manganese is exactly proportioned to the quantity of chlorine they produce when dissolved in hydrochloric acid, and the chlorine can be estimated by the quantity of protosulphate of iron it peroxidises. Of pure peroxide of manganese, 545·9 parts produce 442·6 parts of chlorine, which peroxidise 3456 parts of crystallised protosulphate of iron. Hence 50 grains of peroxide of manganese yield chlorine sufficient to peroxidise 317 grains of protosulphate of iron.

50 grains of the powdered oxide of manganese to be examined are weighed out, and also any known quantity, not less than 317 grains, of sulphate of iron. The oxide of manganese is thrown into a flask containing 1½ ozs. of strong hydrochloric acid, diluted with ½ oz. of water, and a gentle heat applied. The sulphate of iron is gradually added in

small quantities to the acid, so as to absorb the chlorine as it is evolved; and the addition of that salt continued till the liquid, after being heated, gives a blue precipitate with the red prussiate of potash, and has no smell of chlorine, which are indications that the protosulphate of iron is in excess. By weighing what remains of the sulphate of iron, the quantity added is ascertained—say *m* grains. If the whole manganese were peroxide, it would require 317 grains of sulphate of iron, and that quantity would therefore indicate 100 per cent. of peroxide in the specimen; but if a portion of the manganese only is peroxide, it will consume a proportionally small quantity of the sulphate, which quantity will give the proportion of the peroxide, by the proportion as 317 : 100 :: *m* : percentage required. The percentage of peroxide of manganese is thus obtained by multiplying the number of grains of sulphate of iron peroxidised by 0·317. It also follows, that the percentage of chlorine which the same specimen of manganese would afford, is obtained by multiplying the number of grains of sulphate of iron peroxidised by 0·2588.

The quantity of oxygen which any peroxide of manganese loses by becoming protoxide, can be arrived at very exactly and in a very convenient manner, by heating it, in a finely powdered state, with a solution of oxalic acid. The action commences even in the cold; a part of the oxalic acid is converted into carbonic acid, and an oxalate of the protoxide of manganese is formed. Oxalic acid contains 3 atoms of oxygen to 2 atoms of carbon, since carbonic acid contains 4 atoms of oxygen to 2 atoms of carbon: it may be seen that the oxygen which is estimated is equal to one-fourth of that contained in the carbonic acid. The carbonic acid is collected as carbonate of baryta, and the operation performed as follows:—

Place in a small flask 1 part of the pulverised mineral, 4 or 5 parts of oxalic acid, and 10 parts of water; adapt immediately to the matrass a recurved tube of small diameter, placing its open end into a vessel holding about half a pint of saturated baryta water, which must be frequently agitated in order to favour the combination of the evolved carbonic acid with the baryta in solution. When the disengagement of gas nearly ceases, the contents of the flask must be made to boil in order to expel all carbonic acid. It sometimes happens that all the peroxide of manganese assayed is not decomposed by the oxalic acid, which can be ascertained if it has not changed colour, in which case the operation must be repeated.

The following is a method contrived by Dr. Thompson, and is a modification of the one just described. When ordinary care is taken, it is nearly as accurate as assays made in a more expensive manner and with more troublesome apparatus.

Take 50 grains of the finely powdered mineral, and place it in a small flat-bottomed flask (capable of standing the heat of a sand-bath), together with about 1½ ozs. of water, and a ¼ oz. of sulphuric acid. Then place loosely a plug of cotton-wool in the neck to absorb any moisture which the carbonic acid evolved in the course of the experiment might carry over. A tube containing dry chloride of calcium may be adapted to the neck of the flask by means of a perforated cork: this method will ensure greater accuracy. The flask (whether fitted up with the tube or cotton-wool) containing the water, oxide of manganese, and sulphuric acid, is now to be weighed, and 100 grains of oxalic acid placed in it: the tube or wool must be replaced, and the effervescence produced be allowed to proceed as long as it will without the aid of heat: when it ceases, a very gentle heat must be applied for a few minutes, and when cold the flask must be weighed: the loss of weight corresponds to the amount of peroxide present. Thus, supposing

| | |
|---|---|
| The flask, water, peroxide of manganese, sulphuric acid, and tube or wool, weighed . . . . . . | 2000 grs. |
| Oxalic acid added . . . . . . . . . . . | 100 |
| | 2100 |
| And the weight after the operation to be . . . . . | 2060 |
| Loss . . . . . . . | 40 |

The sample under assay would contain 40 grains of peroxide in the 50 grains of ore employed: hence the percentage of pure peroxide would be 80.

In case more exact results are required, the following plan by Fresenius and Will, may be advantageously employed. The description is taken from the English edition.*

The principle upon which this method is based has been applied already by BERTHIER and THOMSON.

The following remarks will serve to explain it.

*a.* If oxalic acid (or an oxalate) is brought into contact with binoxide of manganese, in presence of water and excess of sulphuric acid, protosulphate of manganese is formed, and carbonic acid evolved, while the oxygen, which we may assume to exist in the binoxide of manganese in combination with the protoxide, combines with the elements of the oxalic acid, and thus converts the latter into carbonic acid.

$$MnO_2 + SO_3 + C_2O_3 = MnO,SO_3 + 2CO_2.$$

Each equivalent of available oxygen, or, what amounts to the same, each 1 eq. binoxide of manganese = 43·5, gives 2 eq. carbonic acid = 44.

*b.* If this process is performed in a weighed apparatus from which nothing except the evolved carbonic acid can escape, and which, at the same time, permits the complete expulsion of that acid, the diminution of weight will at once show the amount of carbonic acid which has escaped, and consequently, by a very simple calculation, the quantity of binoxide contained in the analysed manganese ore. As 44 parts by weight of carbonic acid correspond to 43·5 of binoxide of manganese, the carbonic acid found need simply be multiplied by 43·5, and the product divided by 44, or the carbonic acid may be multiplied by

$$\frac{43 \cdot 5}{44} = 0 \cdot 9887$$

to find the corresponding amount of binoxide of manganese.

*c.* But even this calculation may be avoided, by simply using in the operation the exact weight of ore which, if the latter consisted of pure binoxide, would give 100 parts of carbonic acid.

The number of parts evolved of carbonic acid expresses, in that case, directly the number of parts of binoxide contained in 100 parts of the analysed ore. It results from *b* that 98·87 is the number required. Suppose the experiment is made with 0·9887 grm. of the ore, the number of centigrammes of carbonic acid evolved in the process expresses directly the percentage of binoxide contained in the analysed manganese ore. Now, as the amount of carbonic acid evolved from 0·9887 grm. of manganese would be rather small for accurate weighing, it is advisable to take a multiple of this weight, and to divide afterwards the number of centigrammes of carbonic acid evolved from this multiple weight by the same number by which the unit has been multiplied. The multiple which answers the purpose best for superior ores is the triple, = 2·966; for inferior ores, I recommend the quadruple, = 3·955, or the quintuple, = 4·9435.

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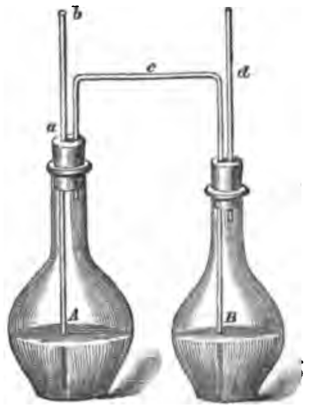
* Fresenius's *Quantitative Analysis*, 4th edition, p. 615. London, Churchills.

The analytical process is performed in the apparatus illustrated in fig. 130.

The flask *A* should hold, up to the neck, about 120 c. c.; *B* about 100 c. c. The latter is half filled with sulphuric acid; the tube *a* is closed at *b* with a little wax ball, or a very small piece of caoutchouc tubing, with a short piece of glass rod inserted in the other end.

Place 2·966, or 3·955, or 4·9435 grms.—according to the quality of the ore—in a watch-glass, and tare the latter most accurately on a delicate balance; then remove the weights from the watch-glass, and replace them by manganese from the tube, very cautiously, with the aid of a gentle tap with the finger, until the equilibrium is exactly restored. Transfer the weighed sample, with the aid of a card, to the flask *A*, add 5—6 grms. neutral oxalate of soda, or about 7·5 grms. neutral oxalate of potassa, in powder, and as much water as will fill the flask to about one-third.

Fig. 130.



Insert the cork into *A*, and tare the apparatus on a strong but delicate balance, by means of shot, and lastly tinfoil, not placed directly on the scale, but in an appropriate vessel. The tare is kept under a glass bell. Try whether the apparatus closes air-tight. Then make some sulphuric acid flow from *B* into *A*, by applying suction to *d*, by means of a caoutchouc tube. The evolution of carbonic acid commences immediately in a steady and uniform manner. When it begins to slacken, cause a fresh portion of sulphuric acid to pass into *A*, and repeat this until the manganese ore is completely decomposed, which, if the sample has been very finely pulverised, requires at the most about five minutes. The complete decomposition of the analysed ore is indicated, on the one hand, by the cessation of the disengagement of carbonic acid, and its non-renewal upon the influx of a fresh portion of sulphuric acid into *A* : and, on the other hand, by the total disappearance of every trace of black powder from the bottom of *A*.*

Now cause some more sulphuric acid to pass from *B* into *A*, to heat the fluid in the latter, and expel the last traces of carbonic acid therein dissolved; remove the wax stopper, or India-rubber tube, from *b*, and apply gentle suction to *d* until the air drawn out tastes no longer of carbonic acid. Let the apparatus cool completely in the air, then place it on the balance, with the tare on the other scale, and restore equilibrium. The number of centigramme weights added, divided by 3, 4, or 5, according to the multiple of 0·9887 grm. used, expresses the percentage of binoxide contained in the analysed ore.

In experiments made with definite quantities of the ore, weighing in an open watch-glass cannot well be avoided, and the dried manganese is thus exposed to the chance of a reabsorption of water from the air, which of course tends to interfere, to however so trifling an extent, with the accuracy of the results. In very precise experiments, therefore, the best way is to analyse an indeterminate quantity of the ore, and to calculate the percentage as shown above. For this purpose, one of the little corked tubes, filled with the dry pulverised ore, is accurately weighed, and about 3 to 5 grms. (according to the quality of the ore) are transferred to the flask *A*. By now reweighing the tube, the exact quantity of ore in the flask is ascertained. To facilitate this operation it is advisable to scratch on the tube, with a file, marks indicating, approximately, the various quantities which may be required for the analysis, according to the quality of the ore.

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* If the manganese ore has been pulverised in an iron mortar, a few black spots (particles of iron from the mortar) will often remain perceptible.

With proper skill and patience on the part of the operator, a good balance, and correct weights, this method gives most accurate and corresponding results, differing in two analyses of the same ore barely to the extent of 0·2 per cent.

If the results of two assays differ by more than 0·2 per cent. a third experiment should be made. In laboratories, where analyses of manganese ores are matters of frequent occurrence, it will be found convenient to use an aspirator for sucking out the carbonic acid. In the case of very moist air, the error which proceeds from the fact that the water in the air drawn through the apparatus is retained, and which is usually quite inconsiderable, may now be increased to an important extent. Under such circumstances connect the end of the tube *b* with a chloride of calcium tube during the suction.

Some ores of manganese contain *carbonates of the alkaline earths*, which of course necessitates a modification of the foregoing process. To ascertain whether carbonates of the alkaline earths are present, boil a sample of the pulverised ore with water, and add nitric acid. If any effervescence takes place, the process is modified as follows (ROHR*) :—

After the weighed portion of ore has been introduced into the flask *A*, treat it with water, so that the flask may be about ¼ full, add a few drops of dilute sulphuric acid (1 part, by weight, sulphuric acid, to 5 parts water) and warm with agitation, preferably in a water-bath. After some time dip a rod in and test whether the fluid possesses a strongly acid reaction. If it does not, add more sulphuric acid. As soon as the whole of the carbonates are decomposed by continued heating of the acidified fluid, completely neutralise the excess of acid with soda solution free from carbonic acid, allow to cool, add the usual quantity of oxalate of soda, and proceed as above.

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* *Zeitschrift f. analyt. Chem.* 1, 48.

If no soda solution free from carbonic acid is at hand, place the oxalate of soda or oxalic acid (about 3 grm.) in a small tube, and suspend this in the flask *A* by means of a thread, fastened by the cork. When the apparatus is tared, and it has been proved to be air-tight, release the thread, and proceed as above.

In the decomposition flask place the ore and some dilute sulphuric acid, and add a solution of oxalic acid through the funnel tube; if necessary, also dilute sulphuric acid. If the ore contains alkaline earthy carbonates, their carbonic acid may be determined in a convenient manner by this process, before the oxidation of the oxalic acid is commenced.

### BLOWPIPE REACTIONS OF MANGANESE.

**SULPHIDE OF MANGANESE.**—*Alone*, in the matrass, undergoes no change.

In the *open tube* roasts slowly, but gives no sublimate. The roasted surface takes a bright green tinge.

*On charcoal*, after complete roasting, behaves with the fluxes like pure oxide of manganese.

**PEROXIDE OF MANGANESE.**—*Alone*, in the matrass, when pure, undergoes no sensible alteration, but in general it contains more or less hydrate of manganese, the water of which may be driven off by means of heat. The more water the heated matter gives off, the less available oxide of manganese it contains. On charcoal it becomes reddish-brown in a good reducing flame.

*With borax and microcosmic salt* it dissolves with a brisk effervescence, produced by disengagement of oxygen; it then behaves as oxide of manganese.

**OXIDE OF MANGANESE.**—*Alone*, the protoxide is not fusible, but becomes brown in a strong flame.

*With borax* it forms a transparent glass, having the colour of amethyst, which becomes colourless in the reducing flame.

If much oxide be present, the glass must be pressed on a cold body, at the instant the blast ceases. The colour returns by a slow cooling.

*With microcosmic salt* it fuses readily, forming a transparent glass, which is colourless in the reducing flame, and amethystine in the oxidising flame. If the glass produced by the union of oxide of manganese with phosphoric acid contain so little of the former as to give no sensible reaction, it can be rendered evident by plunging into the bead a crystal of nitre. The bead swells and foams, and the froth becomes on cooling an amethystine or pale rose tint, according to the quantity present.

*With soda*, the oxide fuses on platinum foil or wire, forming a transparent green glass, which becomes on cooling a bluish-green. This assay is best made on platinum foil. One-thousandth of oxide of manganese gives a very perceptible colour with soda.

# CHAPTER XXII.

## ASSAY OF COBALT AND NICKEL ORES.

ALTHOUGH cobalt and nickel usually accompany each other, yet it will be more convenient to give the ores of both separately, commencing with those of cobalt.

### *Ores of Cobalt.*

Oxide of cobalt, ($CoO$).
Sulphide of cobalt, koboldine ($Co_2S_3$).
Sulphate of cobalt ($CoO,SO_3$).
The arsenides of cobalt.
Arsenio-sulphide, or grey cobalt ($CoAs_2+CoS_2$).
Arsenite of cobalt.

### *Ores of Nickel.*

Oxide of nickel.
Sulphide of nickel.
Arsenide of nickel; kupfernickel.
Arsenio-sulphide of nickel; grey nickel.
Antimonio-sulphide of nickel.
Arseniate of nickel.
Arsenite of nickel.
Silicate of nickel.

*Assay for Cobalt.*—The analysis of cobalt ores is the most tedious, with the exception of those of platinum, of any that fall under the assayer's notice—the greatest difficulty being in the separation of cobalt and nickel. The following process, however, is the most ready that has yet been devised. Very carefully roast, in a porcelain capsule or crucible, 100 or more grains of the sample to be examined. (In case, however, any of the rich ores are under assay, 25 to 50 grains will suffice.) When no more vapours of arsenious acid are evolved, add a little finely-powdered charcoal, and again roast, and so on until no arsenical smell is perceptible. Allow the roasted mass to cool, and then gently heat it in a flask with hydrochloric acid until all but silica is dissolved; evaporate to dryness; allow to cool; moisten with hydrochloric acid; let stand for an hour: then add water, boil, and filter. To the cold filtered solution add a little hydrochloric acid, and pass into this acidulated solution sulphuretted hydrogen gas until in great excess; allow the solution so saturated with gas to remain at rest for two or three hours, then filter it, add a little nitric acid to the filtered solution, and boil so as to peroxidise all the iron present: this point must be carefully attended to, and may be recognised by the addition of a few drops of nitric acid to the hot solution giving no dark tinge. Allow the solution to cool, and if not quite bright, filter it. To the filtered solution add excess of carbonate of baryta. Iron and alumina will be removed after a digestion of three or four hours. Again filter, and to the solution add sulphide of ammonium in excess, gently warm and filter, wash the precipitate, dissolve it in hydrochloric acid; if not bright, filter, and to the filtered solution add cyanide of potassium in excess, and boil. To the boiling solution add a little carbonate of soda —this will precipitate manganese if present—and filter. The solution now contains nothing but cobalt and nickel. These may be separated as follows:—Warm the solution and add to it excess of pulverised peroxide of mercury: this decomposes the potassio-cyanide of nickel, and the whole of the nickel precipitates, the cobalt alone remaining in solution. Remove the nickel by filtration, and neutralise as nearly as possible the filtered solution containing the cobalt by the aid of nitric acid; then add neutral nitrate of mercury solution as long as a white precipitate forms: this precipitate is cyanide of mercury and cobalt. It is collected on a filter, well washed, dried, and then ignited, with free access of atmospheric air, to convert it into black peroxide of cobalt, which is weighed. The nickel precipitate collected on the filter is treated in the same manner: every 100 parts of oxide of nickel correspond to 78·7 parts of metallic nickel. It may be here mentioned, that cobalt is always estimated commercially as oxide, and nickel as metal.

A method of separating these metals, given some years since by Liebig, consists in boiling the mixed double cyanides of nickel and potassium and cobalt and potassium with oxide of mercury. Oxide of nickel is precipitated, while an equivalent quantity of mercury is dissolved as cyanide. The method certainly gives good results, but is not free from objection. Long boiling is necessary before the precipitation is complete, and it is difficult to prevent bumping during ebullition. The excess of oxide of mercury must be separated from the oxide of nickel by a special operation, and the nickel afterwards again precipitated by caustic alkali.

According to Wolcott Gibbs,* these inconveniences may be completely avoided by employing, instead of the oxide alone, a solution of the oxide in the cyanide of mercury. When this solution is added to a hot solution of the double cyanide of nickel and potassium, the whole of the nickel is immediately thrown down as a pale green hydrate of the protoxide. Under the same circumstances cobalt is not precipitated from the double cyanide of cobalt and potassium. Mr. W. N. Hill, who has repeatedly employed this method and carefully tested it, has found that the separation effected is complete. No cobalt can be detected in the precipitated oxide of nickel by the blowpipe, nor can the nickel be detected in the cobalt (finally separated as oxide) by Plattner's process with the gold bead. The solution of oxide of mercury is easily obtained by boiling the oxide with a strong solution of the cyanide, and filtering. According to Kuhn, the cyanide formed in this manner has the formula $HgCy + 3HgO$. The hydrated oxide of nickel precipitated may be filtered off, washed, dried, ignited, and weighed. The cobalt is more readily and conveniently determined by difference, when, as it is always possible, the two metals have been weighed together as sulphates. I am not prepared to say that this modification of Liebig's method of separating nickel and cobalt gives better results than Stromeyer's process by means of nitrite of potassium, but it is at least very much more convenient, and requires much less time. The complete precipitation of cobalt in the form of $Co_2O_3,2NO_3+3KO,NO_3$ usually requires at least forty-eight hours, and rarely succeeds perfectly except in experienced hands.

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* *Chemical News*, March 17, 1865.

M. Terreil has proposed a very excellent method for separating these two metals. The author's method is founded—1, on the insolubility of roseocobaltic hydrochlorate in acid liquids and ammoniacal salts, discovered by M. Fremy; 2, on the rapid transformation of ordinary salts of cobalt into roseocobaltic salts, under the double influence of ammonia and oxidising bodies—such as permanganate of potash and alkaline hypochlorites; 3, on the complete precipitation of manganese in ammoniacal liquids by alkaline hypochlorites, and permanganate of potash.

To separate cobalt from nickel, operate in the following manner:—

To the solution of the two metals add an excess of ammonia, which re-dissolves the two oxides; add to the hot ammoniacal liquid a solution of permanganate of potash, sufficient to cause the liquid to remain coloured violet for a few instants by the excess of permanganate. Boil the liquid for a few minutes, then add a slight excess of hydrochloric acid, to re-dissolve the oxide of manganese which will have formed. Heat the liquid gently for twenty or twenty-five minutes, then let it stand for about twenty-four hours. All the cobalt will then be deposited in the form of a beautiful red-violet crystalline powder; the precipitate is roseocobaltic hydrochlorate, which collect on a weighed filter, wash it on the filter with cold water, then with diluted hydrochloric acid, or with a solution of ammoniacal salt, and then with ordinary alcohol, which frees it from ammoniacal salt. Dry it at 110°, and weigh. 100 parts of roseocobaltic hydrochlorate correspond to 22·761 of metallic cobalt, or to 28·959 of protoxide of cobalt.

It is, however, better to take a given quantity of the roseocobaltic salt, and reduce it by dry hydrogen; this leaves perfectly pure cobalt to be weighed.

Next boil the solution containing nickel to expel the alcohol which has been introduced in washing the cobaltic salt; saturate it with ammonia, add another small excess of permanganate of potash, and boil. All the manganese will be precipitated; filter the liquid, and all the nickel will be found in the filtrate, from which it may easily be separated in the state of sulphide, and then transformed into oxide.

By this process the presence of a ten-thousandth part of cobalt in a salt of nickel may be ascertained.

In this operation an alkaline hypochlorite may take the place of the permanganate of potash, but then the deposit of roseocobaltic salt takes place with extreme slowness, and several days are required to complete it. This reagent is preferable to permanganate when manganese is to be separated from nickel and cobalt.

Should the substance to be analysed contain at the same time cobalt, nickel, and manganese, the latter may be estimated by operating as above, but using given quantities of permanganate and potash estimated beforehand. Lastly, the precipitate of oxide of manganese should be collected, washed, dried, and calcined; from the weight of red oxide obtained, subtract the amount of manganese added in the state of permanganate.

The separation of manganese from cobalt or nickel is extremely easy; it may be effected equally well by means of alkaline hypochlorites or permanganate of potash, which completely precipitate manganese from ammoniacal solutions, and which, under the same conditions, precipitate neither cobalt nor nickel, which remain in the filtered liquids. The method of operating is exactly the same as that above described.

## BLOWPIPE REACTIONS OF COBALT.

**Sulphide of Cobalt.**—In the matrass, gives no volatile substance, and does not decrepitate. In the open tube gives sulphurous acid, and a white sublimate, which consists of drops perceptible by the microscope; they are concentrated sulphuric acid. There are no traces of arsenic.

With the fluxes, the reactions of cobalt so predominate that it is impossible to discover those of iron and copper;

but if it be fused many times with borax, in the exterior flame (that is, the grey bead produced by fusion on charcoal of the mineral itself), the borax removes the cobalt, and the copper concentrates; so that when the mass is fused with microcosmic salt, and exposed to the reducing flame, the red colour of the oxide of copper is produced, tinged, however, by the cobalt blue.

**Arsenical Cobalt.**—*Alone*, in the open tube, gives an abundance of arsenious acid with great facility. In the *matrass*, some species give a little metallic arsenic; others give none.

*On charcoal* all disengage an arsenical smoke and odour, and give by fusion a white metallic bead.

**Cobalt Glance (Tunaberg).**—*Alone*, in the matrass suffers no change.

In the *open tube*, roasts with difficulty, giving no arsenious acid but by a very strong fire, but disengaging sulphurous acid.

*On charcoal*, gives an abundance of fumes, and enters into fusion after some considerable roasting; it then behaves as arsenical cobalt.

**Black Oxide of Cobalt.**—*Alone*, gives a little empyreumatic water.

*On charcoal*, gives traces of arsenic but does not fuse.

Dissolves in borax and microcosmic salt, giving so deep a blue as to disguise all other action.

It is infusible with soda, and generally gives on the platinum wire a mass strongly tinted green by manganese.

**Arseniate of Cobalt.**—*Alone*, in the matrass, gives off water and becomes brown, but furnishes no sublimate.

*On charcoal*, gives off much vapour, and a smell of arsenic. Fuses in a good reducing flame, and is converted into arsenical cobalt.

**Oxide of Cobalt.**—*Alone*, suffers no change.

*With borax* it readily fuses, forming a fine transparent blue glass, which does not become opaque by flaming. A very small quantity colours the glass completely blue, and a large quantity imparts so deep a colour as to make it appear black.

*With microcosmic salt* the appearances are the same as with borax.

*Soda* dissolves but a very small quantity on the platinum wire: the fused mass is pale red by transmitted light, and becomes grey on cooling.

*Carbonate of potash* dissolves a larger quantity of this oxide, forming a black mass, without the slightest mixture of red. This reaction presents a method of distinguishing potash from soda.

The oxide of cobalt is very readily reduced on charcoal in the interior flame, either by an alkali or an alkaline salt. After the soda and charcoal are washed away, a grey metallic powder is obtained, which takes the metallic lustre under the burnisher.

### BLOWPIPE REACTIONS OF NICKEL.

**Sulphide of Nickel.**—In the open tube gives sulphurous acid, becomes black, but does not change form. On charcoal, gives, by aid of a good flame, a mass conglomerated by semi-fusion. It is metallic, malleable, and is pure nickel.

After roasting in the open air, it behaves with fluxes like oxide of nickel.

**Arsenical Nickel**, in the matrass, gives nothing volatile; semifuses at the temperature which softens glass, and a deposit of arsenious acid is formed on the sides of the matrass: this is owing to the included air.

It fuses *on charcoal*, with a vapour and arsenical odour, and a white metallic globule.

In the open tube it roasts easily, with the formation of a large quantity of arsenious acid; the residue is a yellowish-green substance, which, on roasting afresh on charcoal, and fusion with soda and borax, gives a tolerably malleable metallic grain, and is very magnetic.

After roasting, it behaves with the fluxes like oxide of nickel, and generally gives a glass, which is slightly blue, owing to the presence of a small quantity of cobalt.

**Oxide of Nickel.**—*Alone*, is not acted upon.

*With borax* it fuses very readily, producing an orange-yellow or red glass, which, by cooling, becomes yellowish or nearly colourless. A larger quantity of the oxide gives a glass which, when liquid, is deep brown, but which, on cooling, becomes dull red and transparent. This colour is destroyed in the reducing flame, and the glass becomes grey, on account of particles of metallic nickel being disseminated through it.

*With microcosmic salt* it fuses, giving rise to the same phenomena as with borax; but the colour nearly, if not quite, disappears on cooling. It behaves alike in the oxidising and reducing flames, by which reaction it is distinguished from iron. Tin produces, at first, no change; but after a short time the nickel precipitates, and the colour disappears. If cobalt be present, it can then be perceived; but the blue glass is opaque, and cannot be so well distinguished with this flux as when treated in the same manner with borax.

*Soda* does not dissolve oxide of nickel. A large quantity of this flux, however, causes the charcoal to absorb it; it is then reduced, and furnishes, by washing, small, white, brilliant, metallic particles, which are as strongly attracted by the magnet as wrought iron.

The following is Plattner's method for detecting nickel, when contained in large quantities of cobalt:—

Fuse in the oxidising flame a moderate quantity of borax to a bead in the loop of platinum wire, with sufficient oxide of cobalt to give an opaque glass; remove the assay, and prepare one or two similar beads, and place the whole in a charcoal cavity, with a button of pure gold weighing from fifty to eighty milligrammes. The operator must now heat in the reducing flame, until he is satisfied that the whole of the nickel is in a metallic state; the charcoal during the action must be inclined alternately backwards and forwards, so that the gold button may flow through the matter glass, and form an alloy with the reduced particles of nickel. When the golden globule solidifies, it must be extracted with a forceps, placed between paper, and struck with a hammer, so as to detach all the adhering vitreous parts. The auriferous button, which has become more or less grey, from the presence of nickel, and also more brittle than pure gold, is now to be mixed with microcosmic salt, and heated for some time in the oxidating flame. If the borax-glass has not been in the first instance oversaturated with oxide of cobalt, a bead will be now obtained which is coloured only by oxide of nickel, and will therefore appear brownish-red while hot, and when cold reddish-yellow. Should portions of oxide of cobalt be also reduced, as the cobalt is oxidised before the nickel, either a blue glass, coloured by oxide of cobalt, or a green one—if some nickel was also oxidised—will be obtained. In either case the glass must be separated from the button, mixed with more microcosmic salt, and heated in the oxidising flame until it acquires a tinge. If the borax glass had not been oversaturated at the commencement, the colour now obtained will proceed from nickel, although the oxide of cobalt contains a trace only; but if oxide of nickel be not present, the microcosmic bead remains perfectly colourless.

## CHAPTER XXIII.

### THE ASSAY OF SULPHUR.

THE ONLY commercially valuable Sulphur-minerals are:—

I. *Sulphurous Earth* (native sulphur).

In Sicily these minerals are divided into five classes:—

| | | | | | |
|---|---|---|---|---|---|
| 1. | Very rich | ores, | containing | 32—34 | p.c. sulphur |
| 2. | Rich | „ | „ | 24—26 | „ |
| 3. | Good | „ | „ | 16—18 | „ |
| 4. | Middling | „ | „ | 8— 9 | „ |
| 5. | Poor | „ | „ | 3— 5 | „ |

II. *Iron and Copper Pyrites* ($Fe S_2$), ($Cu_2 S, Fe_2 S_3$).

These ores are used to a great extent for the manufacture of sulphuric acid.

In order to approximatively estimate the value of these ores for such manufacture, the following

#### DISTILLING ASSAY

may be used.

A certain quantity of the pulverised ore is heated in a retort, which is furnished with a receiver. The retort may be of glass if the ore is sulphurous earth, but earthen retorts must be employed if pyrites are to be assayed, as the temperature required is much higher (full red heat) than is used for distilling sulphurous earth. If assaying rich pyrites, they must be mixed with quartz sand, as without such mixture a cementation of the ore will take place, which would hinder the sublimation of the sulphur.

The retort is then heated, gradually raising the temperature, till no more sulphur is evolved. The latter will collect in the receiver, which may be, in all cases, of glass, and must be kept cool.

The sulphur derived from sulphurous earth is generally pure, but that from pyrites frequently contains arsenic and selenium, and sometimes traces of thallium.

The manufacture of sulphuric acid is effected by roasting the sulphur-minerals in the presence of air, in some sort of muffle, or in other furnaces. The resulting sulphurous acid is conducted into closed chambers, where it is converted into sulphuric acid.

The yield of sulphur by this process is always larger than that obtained by the above distilling process, and it would be possible to extract by it the whole of the sulphur, or nearly so, if economical considerations did not prevent this, as the separation of the last remaining sulphur requires a disproportionate amount of fuel.

In order to fully ascertain the value of a sulphur ore for the manufacture of sulphuric acid, four assays are required.

1. A determination of the whole amount of sulphur contained in the ore. This must be done by an analysis.

2. A determination of that amount of sulphur which may be obtained by roasting the ore. For this purpose the ore is to be roasted on a small scale, but as nearly as possible as it is done by the manufacturer.

3. A determination of that portion of sulphur which remains in the roasted ore. This must be also done by an analysis.

4. An analysis of the sulphur obtained, as foreign substances contained in it, for instance arsenic, modify its value.

## THE ASSAY OF SULPHUR IN THE WET WAY.

Act upon 50 grains by repeated doses of *aqua regia*, or, better still, strong nitric acid and chlorate of potash, until the ore is entirely decomposed, and if any sulphur remains unacted on, it is quite bright and of a fine amber colour, as described in the Humid Assay of Copper Ores of the Second and Third Classes. When all action has ceased, carefully filter, wash, dry, and weigh the residue; ignite it in a small porcelain dish, weigh again, and the loss of weight will be sulphur. Add to the filtered solution chloride of barium, until no further precipitation takes place; let the whole stand in a warm situation for an hour or so; collect the precipitate on a filter, wash, dry, and ignite it. Every 116 parts of this precipitate of sulphate of baryta correspond to 16 parts of sulphur. The quantity obtained in this manner, added to that obtained in the first part of the operation by the ignition of the insoluble residue, will give the amount of sulphur in the portion of ore operated on.

# CHAPTER XXIV.

## DISCRIMINATION OF GEMS AND PRECIOUS STONES.

In order to explain the introduction of the present chapter into this work, it may be stated, that as many of the precious stones are found in connection with gold, and as the alluvial and other sources of that metal have of late been so wonderfully multiplied, and as diamonds, rubies, emeralds, &c., have by careful examination and research been discovered in Australia and elsewhere, it has been thought advisable to devote a chapter to the elucidation of this important subject; in the hope that, with the instruction here given, those who may cast their lots, either temporarily or permanently, in positions geologically likely to furnish the subjects treated under the present heading, may find themselves materially assisted in the discovery of minerals, on the discrimination of which but little has been popularly written.

The principal sources of recognition are colour, crystalline form, specific gravity, and hardness. In the present chapter will be introduced all the most constantly occurring natural forms of the gems and precious stones mentioned.

The specific gravity or density of a substance is the proportion of its weight to its volume, and it forms a characteristic property of substances. To express the specific gravity in figures it is requisite to compare the density of one substance with that of another, and water at 4° C. is generally adopted as a standard. The specific gravity or density of a substance, therefore, indicates how much a substance is heavier than an equal volume of water.

One cubic centimetre of iron weighs 7·8 grammes, and an equal volume of water = 1 gr.; the specific gravity of iron is, therefore, said to be 7·8.

The specific gravity of a substance may be calculated by dividing its absolute weight by the weight of a corresponding volume of water.

It may be determined at follows, if the sample be of sufficient size to suspend from the pan of a balance by means of a fibre of silk; if not, another mode must be adopted, which will be pointed out as we proceed.

If the mineral can be suspended, attach it by a short fibre of silk to one of the pans of a delicate balance, and ascertain its weight; then immerse it (still suspended to the pan) in distilled water of the temperature 60° Fahr., and then note its weight; it will be found to have lost a certain amount, which will correspond to the weight of the bulk of water it has displaced. Divide its weight in air by the loss of weight in water, and the quotient will be the required specific gravity. This will be more readily understood by an example. Suppose we find the mineral to weigh 80 grs. in the air, and only 66 in water; the loss = 80 — 66 = 14. We must now divide 80 by 14, thus $\frac{80}{14}$ = 5·7, which is about the specific gravity of a sample of bournonite.

We have now the second case to consider; the mineral may be in very small fragments, or it may even be in powder, in which case its specific gravity must be determined thus:—

A small bottle (the ordinary specific gravity bottle is well suited for the purpose), filled with water, is placed on one of the pans of a delicate balance, and its weight ascertained. The mineral in fragments, or even in powder, whose specific gravity is to be determined, is put on the same pan, and the weight of both the filled bottle and the mineral is ascertained.

The mineral is then introduced into the bottle, when part of the water will flow out corresponding to the volume of the mineral, the weight of which may be ascertained by again weighing the bottle, filled partly with water, and partly with mineral.

The following example will suffice to render this clearer:—

| | |
|---|---|
| Suppose the bottle filled with water to weigh . . . . | 500 grs. |
| And the mineral weighing . . . . . . . . | 100 |
| Weight of both . . . . . . | 600 |
| Weight of the bottle when containing the mineral and part of the original water . . . . . . . . | 560 |
| Leaves the absolute weight of the water expelled from the bottle by the mineral . . . . . . . | 40 |
| Therefore the specific gravity of the mineral equals $\frac{100}{40}$ = 2·5 | |

## COLOURLESS STONES.

*The Diamond.*—Specific gravity, 3·48 to 3·52; hardness, 10. The diamond is the hardest of all known substances. The diamond is the only substance which is capable of cutting glass, although most gems will scratch glass; hence it is the utmost term of hardness. When cut and polished, it is the most brilliant gem. It frequently becomes phosphorescent on exposure to light. The greater part of diamonds are limpid and colourless, but many coloured specimens are found; as rose, yellow, orange, blue, green, brown, or even black. It sometimes occurs in regular crystals, octohedrons, dodecahedrons, and more complex forms: see figs. 131, 132, 133, 134.

Fig. 131.

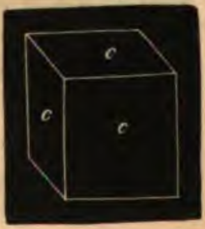


Fig. 132.

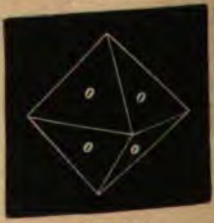


Fig. 133.

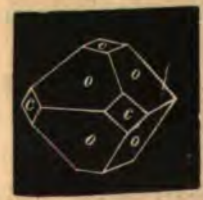
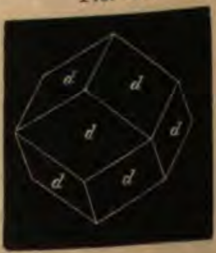


Fig. 134.



The crystalline faces are often curved. The cleavage is octohedral and highly perfect: hence, although diamonds

Fig. 140.

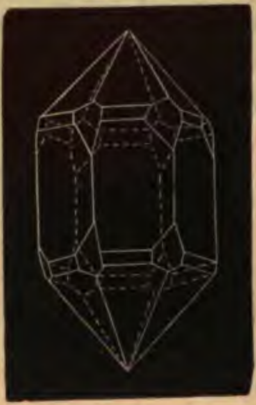
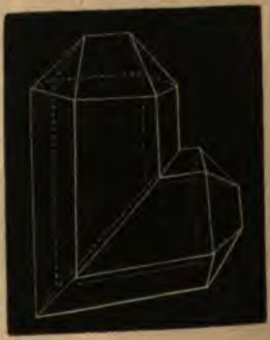


Fig. 141.



Some crystals are as pellucid as glass; others, however, assume all the shades of colour mentioned in the case of the diamond.

Composition ($SiO_3$):—Pure silica or silicic acid.

*White Zircon.*—Specific gravity, 4·44 to 4·8; hardness, 7·5. This stone is often found crystallised in nature in four-sided prisms, terminated by four-sided or rhomboidal or triangular pyramids, and other forms: see figs. 142, 143, 144, 145, 146, 147.

Fig. 142.

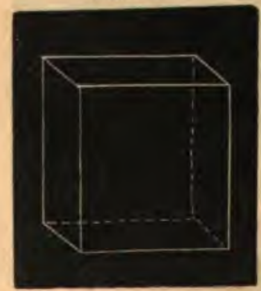


Fig. 143.

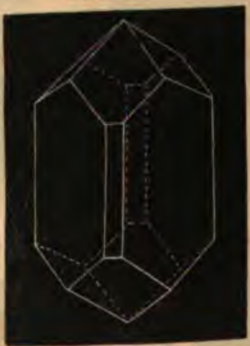
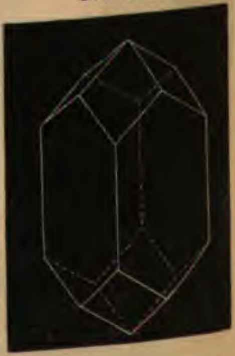


Fig. 144.



These stones are often employed in jewellery under the name of 'rough diamonds.' They often occur brownish-red and brown, red, yellow, and grey: these varieties will be treated under their appropriate heads. It can be readily distinguished from the diamond and quartz by hardness and specific gravity; also by the action of strong hydrochloric acid, which, if dropped on the diamond or quartz, and allowed to remain for a little time, produces no change, but if a zircon be so treated, the spot on which the acid was placed remains dull.

FIG. 145.

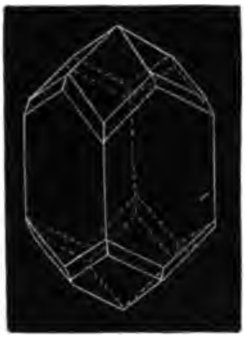


FIG. 146.

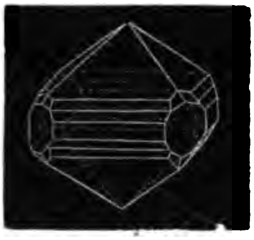
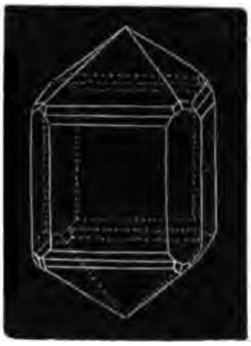


FIG. 147.



Composition $(Zr_2O_3,SiO_3)$:—

| | |
|---|---|
| Zirconia | 67·2 |
| Silicic acid | 33·5 |
| | 100 7 |

*White Sapphire.*—Specific gravity, 3·97 to 4·27; hardness, 9. This stone, in hardness, is next to the diamond. It occurs variously coloured; other colours will be discussed under their appropriate heads. It crystallises in the rhombohedric system, usually in six-sided prisms, but often so very rough as not to be readily distinguishable. May be distinguished by gravity and hardness from all the preceding.

Composition $(Al_2O_3)$:—Pure alumina.

*White Topaz.*—Specific gravity, 3·54; hardness, 9. This variety of topaz, known for its limpidity by the term 'gouttes d'eau,' when polished, has nearly the same lustre as the diamond: the topaz, however, occurs of many colours—see hereafter. It crystallises in the right rectangular prismatic system. The following are some of its natural forms:—figs. 148, 149, 150, 151, 152, 153.

It is readily rendered electric, and retains its electricity for a very considerable time: it is also pyro-electric, or becomes electric when heated,—a property by which it is distinguished from the diamond, its specific gravity being so similar that it cannot be made available as a means of discriminating between the two stones. From the other stones in this group, with the exception of the sapphire, it is readily distinguished by its hardness and gravity, and from the latter by its gravity and pyro-electricity.

FIG. 148.

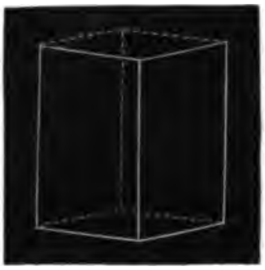


FIG. 149.

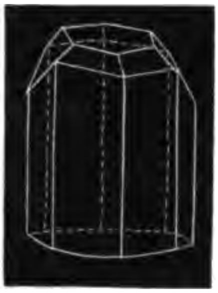


FIG. 150.

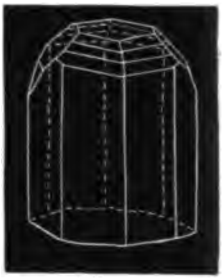


FIG. 151.

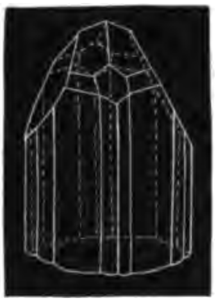


FIG. 152.

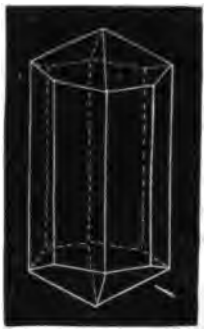
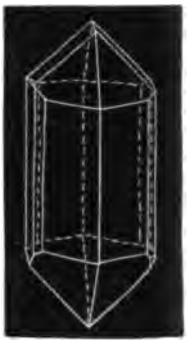


FIG. 153.



Composition :—

| | |
|---|---|
| Silica . . . . . . . . . | 34·2 |
| Alumina . . . . . . . . | 57·5 |
| Fluorine . . . . . . . . | 7·8 |
| | 99·5 |

*Example of the use of Table I.**—A colourless stone, weighing 40 grains in air, is reduced to 24·43 in water. Look in the first column to 40, and then trace along its horizontal line until a number very nearly approaching 24·43 is found ; refer then to the heading of the table, above the number found, and the name there expressed will be that of the stone examined. Supposing, however, the weight of the stone be 41 grains, still the number 24·43 will be the nearest in the table, and ·611 must be added to it, as that sum would be the weight of 41 grains of quartz or water. From the numbers obtained by calculation, also can the specific gravity be determined. If this course be pursued, refer to the bottom line of the table for corresponding number, and to the heading of the table for name of stone. When the weight is any even number of grains (that is, without fractions), the readiest way is to refer to the table (first column), for the number of grains, and then to the horizontal line to corresponding number obtained, which is the weight in water.

---

* The Tables of Comparative Weights were calculated by Brard.

### TABLE I.

### COMPARATIVE TABLE OF THE WEIGHTS OF COLOURLESS STONES WEIGHED IN AIR AND WATER.

| Weight in Air Grains | Weight in Water | | | | |
|---|---|---|---|---|---|
| | White Zircon | White Sapphire | White Topaz | White Diamond | White Quartz |
| 1 | 0·775 | 0·766 | 0·716 | 0·715 | 0·611 |
| 4 | 3·10 | 3·06 | 2·86 | 2·86 | 2·42 |
| 8 | 6·20 | 6·12 | 5·72 | 5·72 | 4·86 |
| 12 | 9·30 | 9·18 | 8·58 | 8·58 | 7·31 |
| 16 | 12·40 | 12·25 | 11·55 | 11·45 | 9·75 |
| 20 | 15·50 | 15·31 | 14·42 | 14·31 | 12·19 |
| 24 | 18·60 | 18·37 | 17·28 | 17·17 | 14·64 |
| 28 | 21·70 | 21·44 | 20·15 | 20·13 | 17·08 |
| 32 | 24·80 | 24·51 | 23·01 | 22·00 | 19·53 |
| 36 | 27·90 | 27·57 | 25·88 | 25·76 | 11·98 |
| 40 | 31·00 | 30·64 | 28·75 | 28·63 | 24·43 |
| 44 | 34·10 | 33·71 | 31·61 | 31·49 | 26·88 |
| 48 | 37·20 | 36·76 | 34·47 | 34·35 | 29·32 |
| 52 | 40·30 | 39·82 | 37·34 | 37·21 | 31·77 |
| 56 | 43·40 | 42·89 | 40·20 | 40·17 | 34·21 |
| 60 | 46·50 | 45·95 | 43·06 | 42·94 | 36·66 |
| 64 | 49·60 | 49·01 | 45·93 | 45·80 | 39·11 |
| 68 | 52·70 | 52·07 | 48·80 | 48·66 | 41·56 |
| 72 | 55·80 | 55·14 | 51·77 | 51·52 | 44·00 |
| 76 | 58·90 | 58·21 | 54·63 | 54·38 | 46·44 |
| 80 | 62·00 | 61·28 | 57·49 | 57·24 | 48·88 |
| 84 | 65·10 | 64·34 | 60·35 | 60·12 | 51·32 |
| 88 | 68·20 | 67·41 | 63·22 | 62·97 | 53·76 |
| 92 | 71·30 | 70·47 | 66·08 | 65·33 | 56·21 |
| 96 | 74·40 | 73·54 | 68·94 | 68·69 | 58·65 |
| 100 | 77·50 | 76·60 | 71·80 | 71·55 | 61·09 |
| Specific Gravity | 4·44 | 4·27 | 3·54 | 3·52 | 2·55 |

Diamond and topaz, however, have very nearly equal density, and a second characteristic must be had recourse to, in order to determine the nature of two stones which have an equal weight in water. This auxiliary character is the development of electricity by heat, a phenomenon exhibited by the topaz but not by the diamond. The test of hardness may be also resorted to.

## YELLOW STONES.

*Yellow Zircon* (*Jargon*).—The crystalline form, characteristics, and composition of this stone have been described under the head 'White Zircon.'

*Yellow Sapphire.*—Characteristics, &c. described under 'White Sapphire.'

*Cymophane* (*Chrysoberyl*).—Specific gravity, 3·65 to 3·89; hardness, 8·5. The cymophane is nearly as hard as the sapphire, harder than the topaz and the emerald: it readily scratches quartz. Its colour is greenish-yellow, and has been placed in the list of yellow stones rather than green, because usually the yellowish tint is most decided. This

Fig. 154.

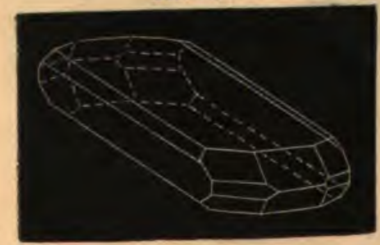


Fig. 155.

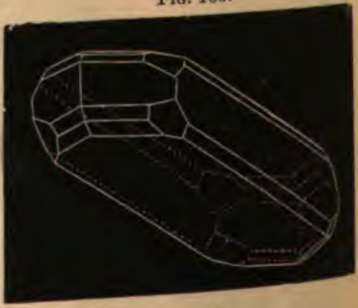


Fig. 156.

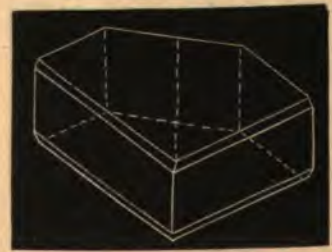
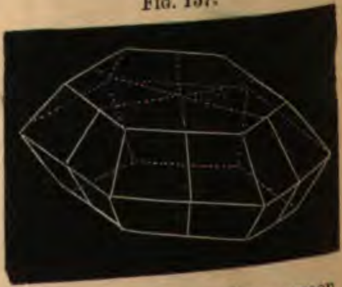


Fig. 157.



tint, which is very agreeable in itself, is often relieved by a small spot of light of a bluish-white tinge, which moves from point to point of the stone as the position of the latter is varied. It is rarely found in regular crystals, but more generally occurs in rolled and rounded masses. For some of its forms, however, see figs. 154, 155, 156, and 157.

Composition :—No. 1 is a sample from the Brazils; No. 2, from Siberia.

| | 1. | 2. |
|---|---|---|
| **Alumina** | 78·10 | 78·92 |
| **Glucina** | 17·94 | 18·02 |
| **Oxide of iron** | 4·46 | 3·12 |
| **Oxide of chromium** | — | 0·36 |
| **Oxides of copper and lead** | — | 0·29 |
| | 100·50 | 100·71 |

*Yellow Topaz.*—The general characteristics of this stone are described under 'White Topaz.'

*Yellow Tourmaline.*—Specific gravity, 3·00—3·22; hardness, 7—7·5. The tourmaline becomes electrical by heat; one portion of a crystal attracts light bodies, the other repels them. Its colour is very varied. The tourmaline has a vitreous fracture. It occurs in semicrystalline prisms of irregular form, generally deeply striated, and in prisms of six or more sides, variously terminated, one end usually differing from the other.

Figs. 158, 159, 160, 161, 162, and 163, represent some of the forms of this mineral.

FIG. 158.

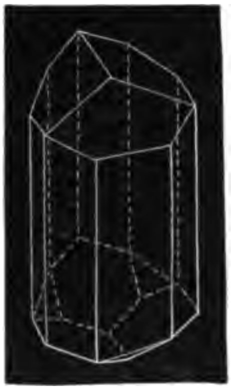


FIG. 159.

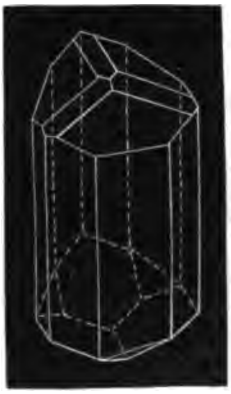


Fig. 160.

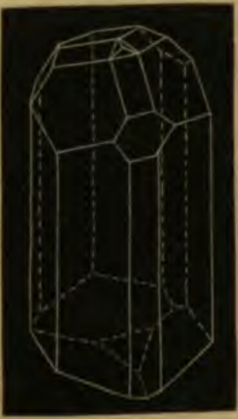


Fig. 161.

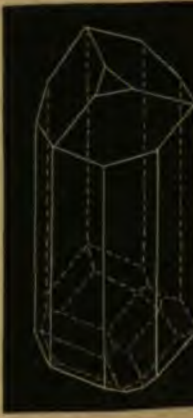


Fig. 162.

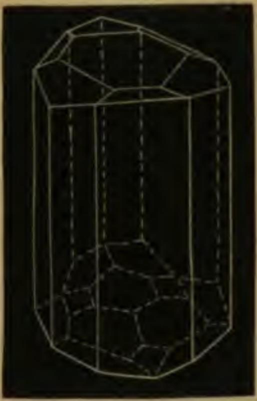
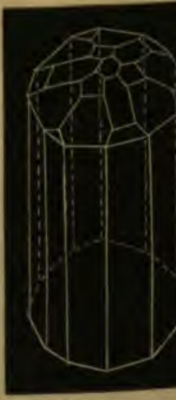


Fig. 163.



*Yellow Emerald.*—Specific gravity, 2·73—2·
ness, 7·5—8. The emerald occurs of many c
tint *par excellence* is green; but there are man
tinged more or less yellow or blue, and they c

Fig. 164.

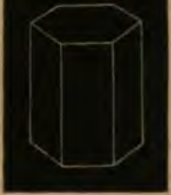


Fig. 165.

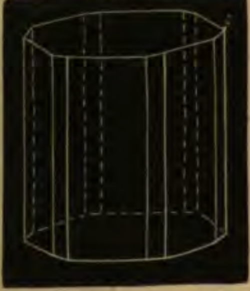
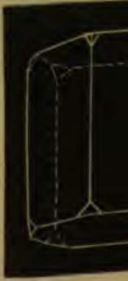


Fig. 1



COMPARATIVE TABLE OF THE WEIGHTS OF YELLOW STONES WEIGHED IN AIR AND WATER.

| Weight in air Grains. | Weights in Water | | | | | | |
|---|---|---|---|---|---|---|---|
| | Yellow Zircon | Yellow Sapphire | Yellow Cymophane | Yellow Topaz | Yellow Tourmaline | Yellow Emerald | Yellow Quartz |
| 1 | 0·775 | 0·766 | 0·738 | 0·716 | 0·690 | 0·633 | 0·611 |
| 4 | 3·10 | 3·06 | 2·95 | 2·86 | 2·76 | 2·53 | 2·42 |
| 8 | 6·20 | 6·12 | 5·90 | 5·72 | 5·52 | 5·06 | 4·86 |
| 12 | 9·30 | 9·18 | 8·85 | 8·58 | 8·28 | 7·59 | 7·31 |
| 16 | 12·40 | 12·25 | 11·80 | 11·55 | 11·04 | 10·12 | 9·75 |
| 20 | 15·50 | 15·31 | 14·75 | 14·42 | 13·80 | 12·65 | 12·19 |
| 24 | 18·60 | 18·07 | 17·70 | 17·28 | 16·56 | 15·19 | 14·04 |
| 28 | 21·70 | 21·44 | 20·65 | 20·15 | 19·32 | 17·72 | 17·08 |
| 32 | 24·80 | 24·51 | 23·60 | 23·01 | 20·08 | 20·25 | 19·53 |
| 36 | 27·90 | 27·57 | 26·55 | 25·88 | 24·84 | 22·77 | 21·98 |
| 40 | 31·00 | 30·64 | 29·50 | 29·75 | 27·60 | 25·30 | 24·43 |
| 44 | 34·10 | 33·71 | 32·45 | 31·61 | 30·36 | 27·83 | 26·88 |
| 48 | 37·20 | 36·76 | 35·40 | 34·47 | 33·12 | 30·36 | 29·32 |
| 52 | 40·30 | 39·82 | 38·35 | 37·34 | 35·88 | 32·89 | 31·77 |
| 56 | 43·40 | 42·89 | 41·30 | 40·20 | 38·64 | 35·43 | 34·21 |
| 60 | 46·50 | 45·95 | 44·25 | 43·06 | 41·40 | 37·94 | 36·66 |
| 64 | 49·60 | 49·01 | 47·20 | 45·93 | 44·16 | 40·47 | 39·11 |
| 68 | 52·70 | 52·08 | 50·15 | 48·90 | 46·92 | 43·00 | 41·56 |
| 72 | 55·80 | 55·14 | 53·10 | 51·77 | 49·68 | 45·53 | 44·00 |
| 76 | 58·90 | 58·21 | 56·05 | 54·63 | 52·44 | 48·07 | 46·44 |
| 80 | 62·00 | 61·28 | 59·00 | 57·49 | 55·20 | 50·60 | 48·88 |
| 84 | 65·10 | 64·34 | 61·95 | 60·35 | 57·96 | 53·13 | 51·32 |
| 88 | 68·20 | 67·41 | 64·90 | 63·22 | 60·72 | 55·66 | 53·76 |
| 92 | 71·30 | 70·47 | 67·85 | 66·08 | 63·48 | 58·19 | 56·21 |
| 96 | 74·40 | 73·54 | 70·80 | 68·94 | 66·24 | 60·72 | 58·65 |
| 100 | 77·50 | 76·60 | 73·75 | 71·80 | 69·00 | 63·25 | 61·09 |
| Specific Gravity | 4·44 | 4·27 | 3·89 | 3·53 | 3·22 | 2·72 | 2·55 |

## BROWN AND FLAME-COLOURED STONES.

*Zircon* (*Hyacinth*).—For characteristics, &c. see 'White Zircon.'

*Vermeil Garnet, Noble Garnet, Almandine.*—Specific gravity, 4—4·2; hardness, 6·5—7·5. There are very many varieties of garnet, variously coloured; but their crystalline form—a rhombic dodecahedron, more or less modified—is a distinguishing characteristic. The colouring matter of the garnet is iron. The following are some of its crystalline forms: figs. 171, 172, 173, 174, and 175:—

Fig. 171.

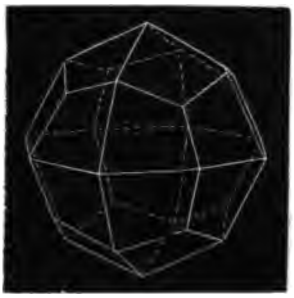


Fig. 172.

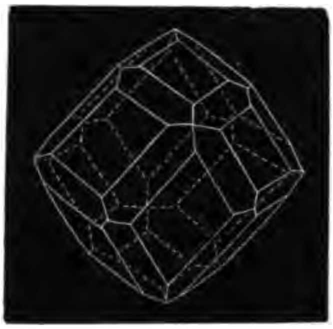


Fig. 173.

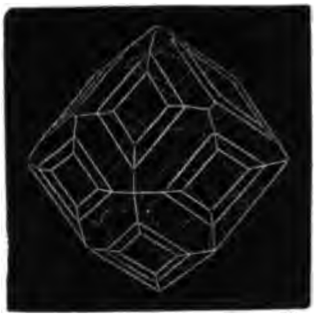


Fig. 174.

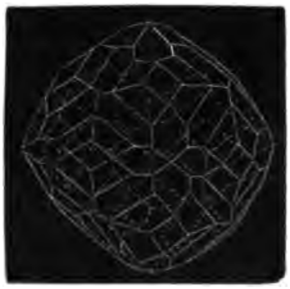
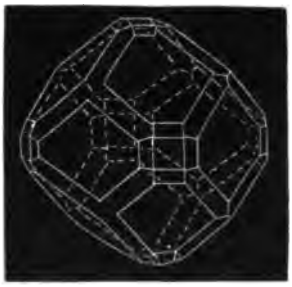


Fig. 175.



Composition :—

| | |
|---|---|
| Silica | 33·75 |
| Alumina | 27·25 |
| Oxide of iron | 36·00 |
| Oxide of manganese | ·25 |
| | 97·25 |

COMPARATIVE TABLE OF THE WEIGHTS OF BROWNISH AND FLAME-COLOURED STONES WEIGHED IN AIR AND WATER.

| Weight in Air Grains | Weight in Water. | | | |
|---|---|---|---|---|
| | Hyacinthine Zircon | Vermeil Garnet | Essonite | Tourmaline |
| 1 | 0·775 | 0·750 | 0·710 | 0·690 |
| 4 | 3·10 | 3·00 | 2·87 | 2·76 |
| 8 | 6·20 | 6·00 | 5·74 | 5·52 |
| 12 | 9·30 | 9·00 | 8·61 | 8·28 |
| 16 | 12·40 | 12·00 | 11·48 | 11·04 |
| 20 | 15·50 | 15·00 | 14·35 | 13·80 |
| 24 | 18·60 | 18·00 | 17·22 | 16·56 |
| 28 | 21·70 | 21·00 | 20·09 | 19·32 |
| 32 | 24·80 | 24·00 | 22·96 | 22·08 |
| 36 | 27·90 | 27·00 | 25·83 | 24·84 |
| 40 | 31·30 | 30 00 | 28·70 | 27·60 |
| 44 | 34·10 | 33·00 | 31·57 | 30·36 |
| 48 | 37·20 | 36·00 | 34·44 | 33·12 |
| 52 | 40·30 | 39·00 | 37·31 | 35·88 |
| 56 | 43·40 | 42·00 | 40·18 | 38·64 |
| 60 | 46·50 | 45·00 | 43·05 | 41·40 |
| 64 | 49·60 | 48·00 | 45·92 | 44·16 |
| 68 | 52·70 | 51·00 | 48·79 | 46·92 |
| 72 | 55·80 | 54·00 | 51·66 | 49·68 |
| 76 | 58·90 | 57·00 | 54·53 | 52·44 |
| 80 | 61·00 | 60·00 | 57·40 | 55·20 |
| 84 | 65·10 | 63·00 | 60·27 | 57·96 |
| 88 | 68·20 | 66·00 | 63·14 | 60·72 |
| 92 | 71·30 | 69·00 | 66·01 | 63·48 |
| 96 | 74·40 | 72·00 | 68·88 | 66·24 |
| 100 | 77·50 | 75·00 | 71·75 | 69·00 |
| Specific Gravity | 4·44 | 4·00 | 3·54 | 3·22 |

*Essonite, Cinnamon Stone.*—Specific gravity, 3·5 to 3·6. This stone has an agreeable orange-yellow tinge, which becomes a warm and brilliant tint when the mass is large. This stone is not usually found crystalline, but in irregular forms and masses, which are characterised by fissures in all directions.

Composition :—

| | |
|---|---|
| Silica . . . . . . . . . | 38·80 |
| Alumina . . . . . . . . . | 21·20 |
| Lime . . . . . . . . . . | 31·25 |
| Oxide of iron with small quantities of Potash and Magnesia . . . . | 6·50 |
| | 97·75 |

*Tourmaline.*—For the characteristics of this mineral see 'Yellow Tourmaline.'

## RED AND ROSE-COLOURED STONES.

*Red Sapphire.*—For characteristics, crystalline form, &c., see 'White Sapphire.'

*Deep Red Garnet, Noble Garnet.*—For characteristics, &c., see 'Vermeil Garnet.'

*Ruby (Spinel).*—Specific gravity, 3·5—3·6; hardness, 8. The ruby readily scratches quartz, but is scratched by the sapphire. Its special colour is red, approaching a rose tint; this tinge, however, undergoes various modifications, such as scarlet, red, rose, yellowish-red, and reddish-purple: it is also found blue and black. Its fracture is flattish-conchoidal, with a splendent vitreous lustre. It occurs crystallised in regular octahedrons, sometimes having their edges replaced as in macles; sometimes it assumes the globular form. The ruby may be distinguished from the red sapphire and the garnet by hardness and specific gravity; and from reddish topaz, which possesses nearly the same specific gravity, by its electric properties.

Composition of red ruby:—

| | |
|---|---|
| Silica | 2·02 |
| Alumina | 69·01 |
| Magnesia | 26·21 |
| Protoxide of iron | 0·71 |
| Oxide of chromium | 1·11 |
| | 99·06 |

*Reddish Topaz.*—For characteristics, &c., see 'White Topaz.'

*Red Tourmaline.*—For characteristics, &c., see 'Yellow Tourmaline.'

### Comparative Table of the Weights of Red or Rose-coloured Stones weighed in Air and Water.

| Weight in Air Grains | Weight in Water | | | | |
|---|---|---|---|---|---|
| | Red Sapphire | Deep Garnets | Rubies | Smoke or Red Topaz | Red Tourmaline |
| 1 | 0·766 | 0·750 | 0·722 | 0·716 | 0·690 |
| 4 | 3·060 | 3·700 | 2·880 | 2·860 | 2·760 |
| 8 | 6·120 | 6·000 | 5·770 | 5·720 | 5·520 |
| 12 | 9·180 | 9·000 | 8·660 | 8·585 | 8·280 |
| 16 | 12·250 | 12·000 | 11·550 | 11·550 | 11·040 |
| 20 | 15·310 | 15·000 | 14·440 | 14·420 | 13·800 |
| 24 | 18·370 | 18·000 | 17·330 | 17·280 | 16·560 |
| 28 | 21·440 | 21·000 | 20·220 | 20·150 | 19·320 |
| 32 | 24·510 | 24·000 | 23·110 | 23·610 | 22·080 |
| 36 | 27·570 | 27·000 | 26·000 | 25·880 | 24·840 |
| 40 | 30·640 | 30 000 | 28·880 | 28·750 | 27·600 |
| 44 | 33·710 | 33·000 | 31·770 | 31·610 | 30·360 |
| 48 | 36·760 | 36·000 | 34·660 | 34·470 | 33·120 |
| 52 | 39·820 | 39·000 | 37·550 | 37·340 | 35·880 |
| 56 | 42·890 | 42·000 | 40·440 | 40·200 | 38·640 |
| 60 | 44·950 | 45·000 | 43·300 | 43·060 | 41·400 |
| 64 | 49·010 | 48·000 | 46·220 | 45·930 | 44·160 |
| 68 | 52·080 | 51·000 | 49·110 | 48·900 | 46·920 |
| 72 | 55·140 | 54·000 | 51·990 | 51·770 | 49·680 |
| 76 | 58·210 | 57·000 | 54·880 | 54·630 | 52·440 |
| 80 | 61·280 | 60·000 | 57·770 | 57·490 | 55·200 |
| 84 | 64 340 | 63·000 | 60·660 | 60·350 | 57·960 |
| 88 | 67·410 | 66·000 | 63·550 | 63·220 | 60·720 |
| 92 | 70·470 | 69·000 | 66·440 | 66·080 | 63·480 |
| 96 | 73·540 | 72·000 | 69·330 | 68·940 | 66·240 |
| 100 | 76·600 | 75·000 | 72·220 | 71·800 | 69·000 |
| Specific Gravity | 4·270 | 4·000 | 3·600 | 3·530 | 3·220 |

## BLUE STONES.

*Blue Sapphire.*—For characteristics, &c., see 'White Sapphire.'

*Disthene, Cyanite.*—Specific gravity, 3·5—3·7; hardness, 5—7. Fine specimens of disthene possess a bright blue colour, which passes insensibly into a deep sky blue. Its transparency is nearly perfect, and it presents small pearly reflections, which add to the beauty of its colour. The primary form of its crystals is a doubly oblique prism, and they cleave very readily in the direction of their length. It can be readily distinguished from the sapphire by its being less hard, as also by its specific gravity. Figs. 176, 177, and 178, represent some of its crystalline forms.

Fig. 176.

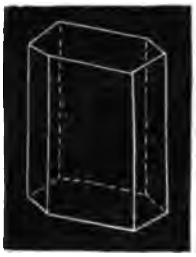


Fig. 177.

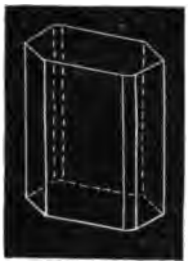
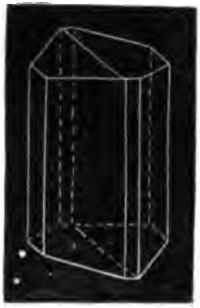


Fig. 178.



Composition of a specimen from St. Gothard :—

| | |
|---|---|
| Silica | 43·0 |
| Alumina | 55·0 |
| Oxide of iron | ·5 |
| | 98·5 |

*Blue Topaz.*—For characteristics, &c., see 'White Topaz.' Blue topaz and disthene having the same specific gravity, may by that test alone be confounded with each other; but the appearance of each is so different, that they can be rarely confounded. If, however, the electrical test be applied, no fear of mistaking one for the other need be entertained, as only the topaz becomes electrical.

*Blue Tourmaline.*—For characteristics, &c. see 'Yellow Tourmaline.'

*Blue Beryl.*—For characteristics, &c., see 'Emerald.' The tint and appearance of this stone and that of the blue topaz are so similar that they cannot be distinguished by that test; their specific gravities, however, are so different, that they may, by this simple means, be readily discriminated.

*Dichroite, Water Sapphire.*—Specific gravity, 2·56—2·65; hardness, 7—7·5. The chief characteristic of this stone is, that it possesses a double colour; that is, it is a fine blue or a normal yellow, as it is viewed in the direction of its base, or the planes of a hexahedral prism, which is its crystalline form. It can be thus readily distinguished, as also by its having nearly the same specific gravity of quartz, and thus being the lightest of the blue stones. Composition :—

| | |
|---|---|
| Silica | 48·35 |
| Alumina | 31·71 |
| Magnesia | 10·16 |
| Protoxide of iron | 8·12 |
| Protoxide of manganese | ·33 |
| Loss in fire (water?) | ·60 |
| | 99·27 |

*Turquoise.* — Specific gravity, 2·8—3; hardness, 5—6. This stone has not been placed in the list of specific gravities, as it can be so readily detected by its appearance. It is bright or greenish-blue in colour; its aspect is earthy or compact. It scratches apatite, and even glass; but is scratched by quartz. It occurs filling fissures, or forming concretions in siliceous and argillo-ferruginous rocks.

### COMPARATIVE TABLE OF THE WEIGHTS OF BLUE STONES WEIGHED IN AIR AND WATER.

| Weight in Air Grains | Weight in Water | | | | | |
|---|---|---|---|---|---|---|
| | Blue Sapphire | Disthene, Cyanite | Blue Topaz | Tourmaline | Blue Beryl | Dichroite, Water Sapphire |
| 1 | 0·766 | 0·717 | 0·716 | 0·690 | 0·633 | 0·622 |
| 4 | 3·06 | 2·87 | 2·86 | 2·16 | 2·53 | 2·49 |
| 8 | 6·12 | 5·74 | 5·72 | 5·52 | 5·06 | 4·98 |
| 12 | 9·18 | 8·61 | 8·58 | 8·28 | 7·59 | 7·47 |
| 16 | 12·25 | 11·48 | 11·45 | 11·04 | 10·12 | 9·96 |
| 20 | 15·31 | 14·35 | 14·42 | 13·80 | 12·65 | 12·45 |
| 24 | 18·37 | 17·22 | 17·18 | 16·56 | 15·19 | 14·94 |
| 28 | 21·44 | 20·09 | 20·05 | 19·32 | 17·72 | 17·43 |
| 32 | 24·51 | 22·96 | 22·91 | 20·08 | 20·25 | 19·92 |
| 36 | 27·57 | 25·83 | 25·78 | 24·84 | 22·77 | 22·41 |
| 40 | 30·64 | 28·70 | 28·65 | 27·60 | 25·30 | 24·90 |
| 44 | 33·71 | 31·57 | 31·51 | 30·36 | 27·83 | 27·39 |
| 48 | 36·76 | 34·44 | 34·37 | 33·12 | 30·36 | 29·88 |
| 52 | 39·82 | 37·31 | 37·24 | 35·88 | 32·89 | 32·37 |
| 56 | 42·89 | 40·18 | 40·10 | 38·64 | 35·43 | 34·86 |
| 60 | 45·95 | 43·05 | 42·96 | 41·40 | 37·94 | 37·35 |
| 64 | 49·01 | 45·92 | 45·83 | 44·16 | 40·47 | 39·84 |
| 68 | 52·08 | 48·79 | 48·80 | 46·92 | 43·00 | 42·33 |
| 72 | 55·14 | 51·66 | 51·67 | 49·68 | 45·53 | 44·82 |
| 76 | 58·21 | 54·53 | 54·53 | 52·44 | 48·07 | 47·31 |
| 80 | 61·28 | 57·40 | 57·40 | 55·20 | 50·60 | 49·80 |
| 84 | 64·34 | 60·27 | 60·25 | 57·96 | 53·13 | 52·29 |
| 88 | 67·41 | 63·14 | 63·12 | 60·72 | 55·66 | 54·78 |
| 92 | 70·47 | 66·01 | 65·98 | 63·48 | 58·19 | 57·27 |
| 96 | 73·54 | 68·88 | 68·84 | 66·24 | 60·72 | 59·76 |
| 100 | 76·60 | 71·75 | 71·70 | 69·00 | 63·25 | 62·25 |
| Specific Gravity | 4·27 | 3·54 | 3·53 | 3·22 | 2·72 | 2·65 |

Composition :—

| | |
|---|---|
| Phosphoric acid . . . . . . | 17·86 |
| Alumina . . . . . . . . | 10·01 |
| Silica . . . . . . . . | 8·90 |
| Peroxide of iron . . . . . . | 36·82 |
| Lime . . . . . . . . . | 0·15 |
| Water and fluoric acid . . . . . | 25·95 |
| | 99·69 |

## VIOLET STONES.

*Violet Sapphire.*—For characteristics, &c., see 'White Sapphire.'

COMPARATIVE TABLE OF THE WEIGHTS OF VIOLET STONES WEIGHED IN AIR AND WATER.

| Weight in Air | Weight in Water | | |
|---|---|---|---|
| Grains | Violet Sapphire | Violet Tourmaline | Amethystine Quartz (Amethyst) |
| 1 | 0·766 | 0·690 | 0·611 |
| 4 | 3·06 | 2·76 | 2·42 |
| 8 | 6·12 | 5·52 | 4·86 |
| 12 | 9·18 | 8·28 | 7·31 |
| 16 | 12·25 | 11·04 | 9·75 |
| 20 | 15·31 | 13·80 | 12·19 |
| 24 | 18·37 | 16·56 | 14·64 |
| 28 | 21·44 | 19·32 | 17·08 |
| 32 | 24·51 | 20·08 | 19·53 |
| 36 | 27·57 | 24·84 | 21·98 |
| 40 | 30·64 | 27·60 | 24·43 |
| 44 | 33·71 | 30·36 | 26·88 |
| 48 | 36·76 | 33·12 | 29·32 |
| 52 | 39·82 | 35·88 | 31·77 |
| 56 | 42·89 | 38·64 | 34·21 |
| 60 | 45·95 | 41·40 | 36·66 |
| 64 | 49·01 | 44·16 | 39·11 |
| 68 | 52·02 | 46·92 | 41·56 |
| 72 | 55·14 | 49·68 | 44·00 |
| 76 | 58·21 | 52·44 | 46·44 |
| 80 | 61·28 | 55·20 | 48·88 |
| 84 | 64·34 | 57·96 | 51·32 |
| 88 | 67·41 | 60·72 | 53·76 |
| 92 | 70·47 | 63·48 | 56·21 |
| 96 | 73·54 | 66·24 | 58·65 |
| 100 | 76·60 | 69·00 | 61·09 |
| Specific Gravity | 4·27 | 3·22 | 2·55 |

*Violet Tourmaline.*—For characteristics, &c., see 'Yellow Tourmaline.'

*Violet Quartz, Amethyst.*—For characteristics, &c., see 'White Quartz.'

## GREEN STONES.

*Green Sapphire.*—For characteristics, &c., see 'Yellow Emerald.'

*Peridot, Crysolite.*—Specific gravity, 3·3—3·5; hardness, 6·5—7. This stone has a more or less deep olive or yellowish-green colour. It is more generally found in rolled grains than in regular prismatic crystals. It is possessed in a very high degree of double refraction. Figs. 179, 180, 181, and 182, represent some of its crystalline forms.

FIG. 179.

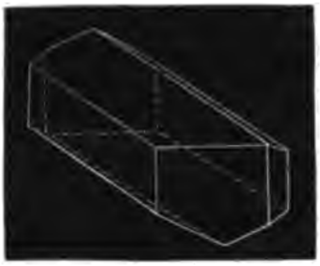


FIG. 180.

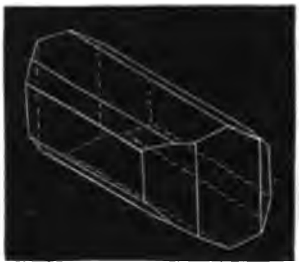


FIG. 181.

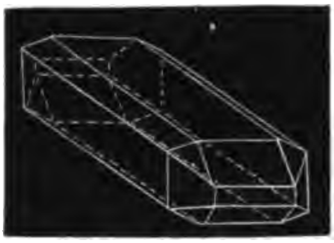
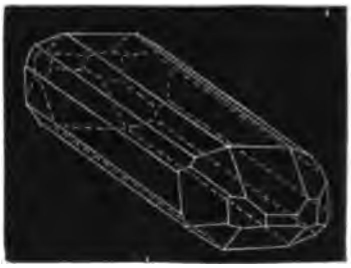


FIG. 182.



*Green Tourmaline.*—For characteristics, see 'Yellow Tourmaline.'

*Emerald.*—For characteristics see 'Yellow Emerald.'

*Aqua-marine.*—This stone possesses a very pale green tinge. For other characteristics, see 'Yellow Emerald.'

*Chrysoprase.*—This mineral is a green-coloured quartz, and can be readily recognised by referring to the characteristics of quartz.

### COMPARATIVE TABLE OF THE WEIGHTS OF GREEN STONES WEIGHED IN AIR AND WATER.

| Weight in Air Grains | Weight in Water. | | | | | |
|---|---|---|---|---|---|---|
| | Green Sapphire | Peridot | Green Tourmaline | Emerald | Aqua Marine | Chryso-prase |
| 1 | 0·766 | 0·708 | 0·690 | 0·633 | 0·633 | 0·611 |
| 4 | 3·06 | 2·83 | 2·76 | 2·53 | 2·53 | 2·42 |
| 8 | 6·12 | 5·66 | 5·52 | 5·06 | 5·06 | 4·86 |
| 12 | 9·18 | 8·49 | 8·28 | 7 59 | 7·59 | 7·31 |
| 16 | 12·25 | 11·32 | 11·04 | 10·12 | 10·12 | 9·75 |
| 20 | 15·31 | 14·16 | 13·80 | 12·65 | 12·65 | 12·19 |
| 24 | 18·37 | 16·99 | 16·56 | 15·19 | 15·19 | 14·64 |
| 28 | 21·44 | 19·82 | 19·32 | 17·72 | 17·72 | 17·08 |
| 32 | 24·51 | 22·65 | 22·08 | 20·25 | 20·25 | 19 53 |
| 36 | 27·57 | 25·48 | 24·84 | 22·77 | 27·77 | 21·98 |
| 40 | 30·64 | 28·32 | 27·60 | 25·30 | 25·30 | 24·43 |
| 44 | 33·71 | 31·15 | 30·36 | 27·83 | 27·83 | 36·88 |
| 48 | 36·76 | 33·98 | 33·12 | 30·36 | 30·36 | 29·32 |
| 52 | 39·82 | 36·81 | 35·88 | 32·89 | 32·89 | 31·77 |
| 56 | 42·89 | 39·64 | 38·64 | 35·43 | 35·43 | 34·21 |
| 60 | 45·95 | 42·48 | 41·40 | 37·94 | 37·94 | 36·66 |
| 64 | 49·01 | 45·31 | 44·16 | 40·47 | 40·47 | 39·11 |
| 68 | 52·08 | 48·14 | 46·92 | 43·00 | 43·00 | 41·56 |
| 72 | 55·14 | 50·97 | 49·68 | 45·53 | 45·53 | 44·00 |
| 76 | 58·21 | 53·80 | 52·44 | 48·07 | 48·07 | 46·44 |
| 80 | 61·28 | 56·64 | 55·20 | 50·60 | 50·60 | 48·88 |
| 84 | 64·34 | 59·47 | 57 96 | 53·13 | 53·13 | 51·32 |
| 88 | 67·41 | 62·30 | 60·72 | 55·66 | 55·66 | 53·76 |
| 92 | 70·47 | 65·13 | 63·48 | 58·19 | 58·19 | 56·21 |
| 96 | 73·54 | 67·96 | 66·24 | 60·72 | 60·72 | 58·65 |
| 100 | 76·60 | 70·80 | 69·00 | 63·25 | 63·25 | 61·09 |
| Specific Gravity } | 4·27 | 3·42 | 3·22 | 2·72 | 2·72 | 2·56 |

### STONES POSSESSING A PLAY OF COLOURS (CHATOYANT).

In the following list of stones no regard has been paid to absolute colours, but only to the play of colours the stones exhibit. This play or reflection is of two kinds: in some, as the sapphires, it appears as a white star with six rays, on a blue, red, or yellow ground; or on a purple ground in the garnet. In others it is but a point or mass of pearly light, which sometimes appears to occupy the whole of the stone, and varies according to the inclination given to the stone. The cymophane, crysolite quartz, Egyptian emerald, felspar, and cat's eye, belong to this class.

The specific gravities of such stones as the opal, &c.,

have not been given, as the appearance sufficiently characterises them.

*Sapphire.*—For characteristics, &c., see 'White Sapphire.'

*Garnet.*—For characteristics, &c., see 'Vermeil Garnet.'

*Cymophane.*—See 'Cymophane.'

*Antique Emerald.*—For characteristics, &c., see 'Yellow Emerald.'

*Quartz.*—See 'White Quartz.'

*Felspar, Nacreous Felspar, Fish-eye, &c.* — Specific gravity, 2·3—2·5; hardness, 4·5—5. This species of felspar has a lamellar texture. It will be seen by the lowness of its specific gravity that it cannot be readily confounded with other stones. In appearance its transparency is nebulous, and it presents pearly white reflections, which float

FIG. 183.

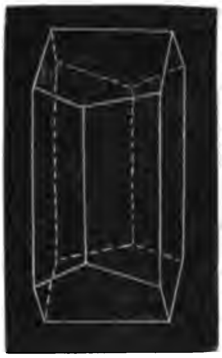


FIG. 184.

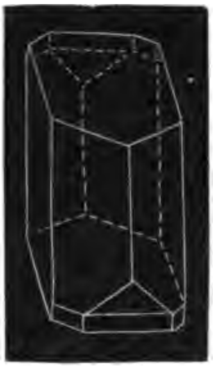


FIG. 185.

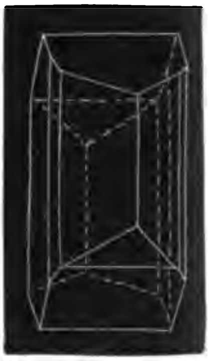


FIG. 186.

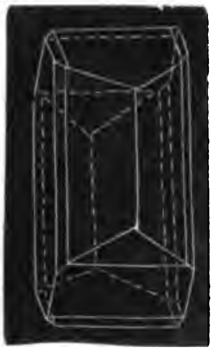


FIG. 187.

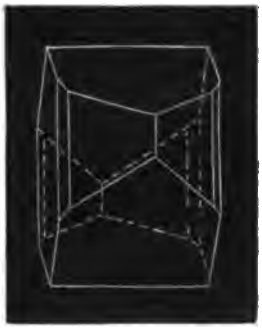
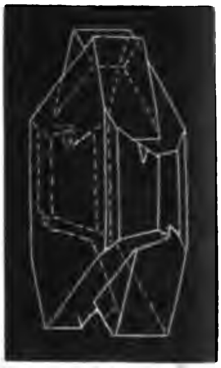


FIG. 188.



about and vacillate in proportion as its position changes. The foregoing are some of the forms of felspar ; see figs. 183, 184, 185, 186, 187, and 188.

Composition :—

| | |
|---|---|
| Potash | 5·26 |
| Silica | 52·90 |
| Lime | 25·20 |
| Water | 16·00 |
| Fluoric acid | 0·82 |
| | 100·18 |

COMPARATIVE TABLE OF THE WEIGHTS OF STONES POSSESSING A PLAY OF COLOURS (CHATOYANT).

| Weight in Air Grains | Weight in Water | | | | | |
|---|---|---|---|---|---|---|
| | Sapphires | Garnets | Cymophane | Antique Emerald | Quartz | Felspar |
| 1 | 0·766 | 0·750 | 0·738 | 0·633 | 0·611 | 0·592 |
| 4 | 3·06 | 3·00 | 2·95 | 2·53 | 2·42 | 2·37 |
| 8 | 6·12 | 6·00 | 5·90 | 5·06 | 4·86 | 4·74 |
| 12 | 9·18 | 9·00 | 8·85 | 7·59 | 7·31 | 7·11 |
| 16 | 12·25 | 12·00 | 11·80 | 10·12 | 9·75 | 9·47 |
| 20 | 15·31 | 15·00 | 14·75 | 12·65 | 12·19 | 11·84 |
| 24 | 18·37 | 18·00 | 17·70 | 15·19 | 14·64 | 14·20 |
| 28 | 21·44 | 21·00 | 20·65 | 17·72 | 17·08 | 16·57 |
| 32 | 24·51 | 24·00 | 23·60 | 20·25 | 19·53 | 18·94 |
| 36 | 27·57 | 27·00 | 26·55 | 22·77 | 21·98 | 21·31 |
| 40 | 30·64 | 30·00 | 29·50 | 25·30 | 24·43 | 23·68 |
| 44 | 33·71 | 33·00 | 32·46 | 27·83 | 26·88 | 26·05 |
| 48 | 36·76 | 36·00 | 35·40 | 30·36 | 29·32 | 28·42 |
| 52 | 39·84 | 39·00 | 38·35 | 32·89 | 31·77 | 30·79 |
| 56 | 42·89 | 42·00 | 41·30 | 35·43 | 34·21 | 33·15 |
| 60 | 45·95 | 45·00 | 44·25 | 37·94 | 36·66 | 35·52 |
| 64 | 49·01 | 48·00 | 47·20 | 40·47 | 39·11 | 37·88 |
| 68 | 52·07 | 51·00 | 50·15 | 43·00 | 41·56 | 40·25 |
| 72 | 55·14 | 54·00 | 53·10 | 45·53 | 44·00 | 42·62 |
| 76 | 58·21 | 57·00 | 56·05 | 48·07 | 46·44 | 44·99 |
| 80 | 61·28 | 60·00 | 59·00 | 50·60 | 48·88 | 47·36 |
| 84 | 64·34 | 63·00 | 61·95 | 53·13 | 51·32 | 49·73 |
| 88 | 67·47 | 66·00 | 64·90 | 55·66 | 53·76 | 52·10 |
| 92 | 70·47 | 69·00 | 67·85 | 58·19 | 56·21 | 54·47 |
| 96 | 73·54 | 72·00 | 70·80 | 60·72 | 58·65 | 56·84 |
| 100 | 76·60 | 75·00 | 73·75 | 63·25 | 61·09 | 59·21 |
| Specific Gravity | 4·27 | 4·00 | 3·89 | 2·72 | 2·55 | 2·45 |

# APPENDIX.

## TABLE I.

Showing the Quantity of Fine Gold in 1 oz. of any Alloy to ⅛ of a Carat Grain and the Mint Value of 1 oz. of each Alloy.

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 1 | 0 | 0·000 | 24 | 0 | 0 | 4 | 4 | 11·4545 =$19.60 |
| 0 | 19 | 23·375 | 23 | 3 | 7 | 4 | 4 | 10·1271 |
| 0 | 19 | 22·750 | 23 | 3 | 6 | 4 | 4 | 8·7997 |
| 0 | 19 | 22·125 | 23 | 3 | 5 | 4 | 4 | 7·4723 |
| 0 | 19 | 21·500 | 23 | 3 | 4 | 4 | 4 | 6·1448 |
| 0 | 19 | 20·875 | 23 | 3 | 3 | 4 | 4 | 4·8174 |
| 0 | 19 | 20·250 | 23 | 3 | 2 | 4 | 4 | 3·4900 |
| 0 | 19 | 19·625 | 23 | 3 | 1 | 4 | 4 | 2·1626 |
| 0 | 19 | 19·000 | 23 | 3 | 0 | 4 | 4 | 0·8352 |
| 0 | 19 | 18·375 | 23 | 2 | 7 | 4 | 3 | 11·5078 |
| 0 | 19 | 17·750 | 23 | 2 | 6 | 4 | 3 | 10·1804 |
| 0 | 19 | 17·125 | 23 | 2 | 5 | 4 | 3 | 8·8529 |
| 0 | 19 | 16·500 | 23 | 2 | 4 | 4 | 3 | 7·5255 |
| 0 | 19 | 15·875 | 23 | 2 | 3 | 4 | 3 | 6·1981 |
| 0 | 19 | 15·250 | 23 | 2 | 2 | 4 | 3 | 4·8707 |
| 0 | 19 | 14·625 | 23 | 2 | 1 | 4 | 3 | 3·5433 |
| 0 | 19 | 14·000 | 23 | 2 | 0 | 4 | 3 | 2·2159 |
| 0 | 19 | 13·375 | 23 | 1 | 7 | 4 | 3 | 0·8885 |
| 0 | 19 | 12·750 | 23 | 1 | 6 | 4 | 2 | 11·5610 |
| 0 | 19 | 12·125 | 23 | 1 | 5 | 4 | 2 | 10·2336 |
| 0 | 19 | 11·500 | 23 | 1 | 4 | 4 | 2 | 8·9062 |
| 0 | 19 | 10·875 | 23 | 1 | 3 | 4 | 2 | 7·5788 |
| 0 | 19 | 10·250 | 23 | 1 | 2 | 4 | 2 | 6·2514 |
| 0 | 19 | 9·625 | 23 | 1 | 1 | 4 | 2 | 4·9240 |
| 0 | 19 | 9·000 | 23 | 1 | 0 | 4 | 2 | 3·5965 |
| 0 | 19 | 8·375 | 23 | 0 | 7 | 4 | 2 | 2·2691 |
| 0 | 19 | 7·750 | 23 | 0 | 6 | 4 | 2 | 0·9417 |
| 0 | 19 | 7·125 | 23 | 0 | 5 | 4 | 1 | 11·6143 |
| 0 | 19 | 6·500 | 23 | 0 | 4 | 4 | 1 | 10·2869 |
| 0 | 19 | 5·875 | 23 | 0 | 3 | 4 | 1 | 8·9595 |
| 0 | 19 | 5·250 | 23 | 0 | 2 | 4 | 1 | 7·6321 |
| 0 | 19 | 4·625 | 23 | 0 | 1 | 4 | 1 | 6·3047 |
| 0 | 19 | 4·000 | 23 | 0 | 0 | 4 | 1 | 4·9772 |
| 0 | 19 | 3·375 | 22 | 3 | 7 | 4 | 1 | 3·6498 |
| 0 | 19 | 2·750 | 22 | 3 | 6 | 4 | 1 | 2·3224 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 19 | 2·125 | 22 | 3 | 5 | 4 | 1 | 0·9950 |
| 0 | 19 | 1·500 | 22 | 3 | 4 | 4 | 0 | 11·6676 |
| 0 | 19 | 0·875 | 22 | 3 | 3 | 4 | 0 | 10·3402 |
| 0 | 19 | 0·250 | 22 | 3 | 2 | 4 | 0 | 8·0127 |
| 0 | 18 | 23·625 | 22 | 3 | 1 | 4 | 0 | 7·6854 |
| 0 | 18 | 23·000 | 22 | 3 | 0 | 4 | 0 | 6·3579 |
| 0 | 18 | 22·375 | 22 | 2 | 7 | 4 | 0 | 4·0305 |
| 0 | 18 | 21·750 | 22 | 2 | 6 | 4 | 0 | 3·7031 |
| 0 | 18 | 21·125 | 22 | 2 | 5 | 4 | 0 | 2·3757 |
| 0 | 18 | 20·500 | 22 | 2 | 4 | 4 | 0 | 0·0482 |
| 0 | 18 | 19·875 | 22 | 2 | 3 | 3 | 19 | 11·7208 |
| 0 | 18 | 19·250 | 22 | 2 | 2 | 3 | 19 | 10·3934 |
| 0 | 18 | 18·625 | 22 | 2 | 1 | 3 | 19 | 8·0660 |
| 0 | 18 | 18·000 | 22 | 2 | 0 | 3 | 19 | 7·7386 |
| 0 | 18 | 17·375 | 22 | 1 | 7 | 3 | 19 | 6·4112 |
| 0 | 18 | 16·750 | 22 | 1 | 6 | 3 | 19 | 4·0838 |
| 0 | 18 | 16·125 | 22 | 1 | 5 | 3 | 19 | 3·7563 |
| 0 | 18 | 15·500 | 22 | 1 | 4 | 3 | 19 | 2·4289 |
| 0 | 18 | 14·875 | 22 | 1 | 3 | 3 | 19 | 0·1015 |
| 0 | 18 | 14·250 | 22 | 1 | 2 | 3 | 18 | 11·7741 |
| 0 | 18 | 13·625 | 22 | 1 | 1 | 3 | 18 | 10·4467 |
| 0 | 18 | 13·000 | 22 | 1 | 0 | 3 | 18 | 8·1193 |
| 0 | 18 | 12·375 | 22 | 0 | 7 | 3 | 18 | 7·7919 |
| 0 | 18 | 11·750 | 22 | 0 | 6 | 3 | 18 | 6·4644 |
| 0 | 18 | 11·125 | 22 | 0 | 5 | 3 | 18 | 4·1370 |
| 0 | 18 | 10·500 | 22 | 0 | 4 | 3 | 18 | 3·8096 |
| 0 | 18 | 9·875 | 22 | 0 | 3 | 3 | 18 | 2·4822 |
| 0 | 18 | 9·250 | 22 | 0 | 2 | 3 | 18 | 0·1548 |
| 0 | 18 | 8·625 | 22 | 0 | 1 | 3 | 17 | 11·8274 |
| 0 | 18 | 8·000 | 22 | 0 | 0 | 3 | 17 | 10·5000 |
| 0 | 18 | 7·375 | 21 | 3 | 7 | 3 | 17 | 8·1725 |
| 0 | 18 | 6·750 | 21 | 3 | 6 | 3 | 17 | 7·8451 |
| 0 | 18 | 6·125 | 21 | 3 | 5 | 3 | 17 | 6·5177 |
| 0 | 18 | 5·500 | 21 | 3 | 4 | 3 | 17 | 4·1903 |
| 0 | 18 | 4·875 | 21 | 3 | 3 | 3 | 17 | 3·8629 |
| 0 | 18 | 4·250 | 21 | 3 | 2 | 3 | 17 | 2·5355 |
| 0 | 18 | 3·625 | 21 | 3 | 1 | 3 | 17 | 0·2081 |
| 0 | 18 | 3·000 | 21 | 3 | 0 | 3 | 16 | 11·8806 |
| 0 | 18 | 2·375 | 21 | 2 | 7 | 3 | 16 | 10·5532 |
| 0 | 18 | 1·750 | 21 | 2 | 6 | 3 | 16 | 8·2258 |
| 0 | 18 | 1·125 | 21 | 2 | 5 | 3 | 16 | 7·8984 |
| 0 | 18 | 0·500 | 21 | 2 | 4 | 3 | 16 | 6·5710 |
| 0 | 17 | 23·875 | 21 | 2 | 3 | 3 | 16 | 4·2436 |
| 0 | 17 | 23·250 | 21 | 2 | 2 | 3 | 16 | 3·9162 |

| **Fine Gold,** Per Ounce | | | **Carat Gold,** Per Ounce | | | **Sterling Value,** Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 17 | 22·625 | 21 | 2 | 1 | 3 | 16 | 2·5887 |
| 0 | 17 | 22·000 | 21 | 2 | 0 | 3 | 16 | 1·2613 |
| 0 | 17 | 21·375 | 21 | 1 | 7 | 3 | 15 | 11·9339 |
| 0 | 17 | 20·750 | 21 | 1 | 6 | 3 | 15 | 10·6065 |
| 0 | 17 | 20·125 | 21 | 1 | 5 | 3 | 15 | 9·2791 |
| 0 | 17 | 19·500 | 21 | 1 | 4 | 3 | 15 | 7·9517 |
| 0 | 17 | 18·875 | 21 | 1 | 3 | 3 | 15 | 6·6243 |
| 0 | 17 | 18·250 | 21 | 1 | 2 | 3 | 15 | 5·2968 |
| 0 | 17 | 17·625 | 21 | 1 | 1 | 3 | 15 | 3·9694 |
| 0 | 17 | 17·000 | 21 | 1 | 0 | 3 | 15 | 2·6420 |
| 0 | 17 | 16·375 | 21 | 0 | 7 | 3 | 15 | 1·3146 |
| 0 | 17 | 15·750 | 21 | 0 | 6 | 3 | 14 | 11·9872 |
| 0 | 17 | 15·125 | 21 | 0 | 5 | 3 | 14 | 10·6598 |
| 0 | 17 | 14·500 | 21 | 0 | 4 | 3 | 14 | 9·3324 |
| 0 | 17 | 13·875 | 21 | 0 | 3 | 3 | 14 | 8·0049 |
| 0 | 17 | 13·250 | 21 | 0 | 2 | 3 | 14 | 6·6775 |
| 0 | 17 | 12·625 | 21 | 0 | 1 | 3 | 14 | 5·3501 |
| 0 | 17 | 12·000 | 21 | 0 | 0 | 3 | 14 | 4·0227 |
| 0 | 17 | 11·375 | 20 | 3 | 7 | 3 | 14 | 2·6953 |
| 0 | 17 | 10·750 | 20 | 3 | 6 | 3 | 14 | 1·3678 |
| 0 | 17 | 10·125 | 20 | 3 | 5 | 3 | 14 | 0·0404 |
| 0 | 17 | 9·500 | 20 | 3 | 4 | 3 | 13 | 10·7130 |
| 0 | 17 | 8·875 | 20 | 3 | 3 | 3 | 13 | 9·3856 |
| 0 | 17 | 8·250 | 20 | 3 | 2 | 3 | 13 | 8·0582 |
| 0 | 17 | 7·625 | 20 | 3 | 1 | 3 | 13 | 6·7308 |
| 0 | 17 | 7·000 | 20 | 3 | 0 | 3 | 13 | 5·4034 |
| 0 | 17 | 6·375 | 20 | 2 | 7 | 3 | 13 | 4·0759 |
| 0 | 17 | 5·750 | 20 | 2 | 6 | 3 | 13 | 2·7485 |
| 0 | 17 | 5·125 | 20 | 2 | 5 | 3 | 13 | 1·4211 |
| 0 | 17 | 4·500 | 20 | 2 | 4 | 3 | 13 | 0·0937 |
| 0 | 17 | 3·875 | 20 | 2 | 3 | 3 | 12 | 10·7663 |
| 0 | 17 | 3·250 | 20 | 2 | 2 | 3 | 12 | 9·4389 |
| 0 | 17 | 2·625 | 20 | 2 | 1 | 3 | 12 | 8·1115 |
| 0 | 17 | 2·000 | 20 | 2 | 0 | 3 | 12 | 6·7840 |
| 0 | 17 | 1·375 | 20 | 1 | 7 | 3 | 12 | 5·4566 |
| 0 | 17 | 0·750 | 20 | 1 | 6 | 3 | 12 | 4·1292 |
| 0 | 17 | 0·125 | 20 | 1 | 5 | 3 | 12 | 2·8018 |
| 0 | 16 | 23·500 | 20 | 1 | 4 | 3 | 12 | 1·4744 |
| 0 | 16 | 22·875 | 20 | 1 | 3 | 3 | 12 | 0·1470 |
| 0 | 16 | 22·250 | 20 | 1 | 2 | 3 | 11 | 10·8196 |
| 0 | 16 | 21·625 | 20 | 1 | 1 | 3 | 11 | 9·4921 |
| 0 | 16 | 21·000 | 20 | 1 | 0 | 3 | 11 | 8·1647 |
| 0 | 16 | 20·375 | 20 | 0 | 7 | 3 | 11 | 6·8373 |
| 0 | 16 | 19·750 | 20 | 0 | 6 | 3 | 11 | 5·5099 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£.* | *s.* | *d.* |
| 0 | 16 | 19·125 | 20 | 0 | 5 | 3 | 11 | 4·1825 |
| 0 | 16 | 18·500 | 20 | 0 | 4 | 3 | 11 | 2·8551 |
| 0 | 16 | 17·875 | 20 | 0 | 3 | 3 | 11 | 1·5277 |
| 0 | 16 | 17·250 | 20 | 0 | 2 | 3 | 11 | 0·2002 |
| 0 | 16 | 16·625 | 20 | 0 | 1 | 3 | 10 | 10·8728 |
| 0 | 16 | 16·000 | 20 | 0 | 0 | 3 | 10 | 9·5454 |
| 0 | 16 | 15·375 | 19 | 3 | 7 | 3 | 10 | 8·2180 |
| 0 | 16 | 14·750 | 19 | 3 | 6 | 3 | 10 | 6·8906 |
| 0 | 16 | 14·125 | 19 | 3 | 5 | 3 | 10 | 5·5632 |
| 0 | 16 | 13·500 | 19 | 3 | 4 | 3 | 10 | 4·2357 |
| 0 | 16 | 12·875 | 19 | 3 | 3 | 3 | 10 | 2·9083 |
| 0 | 16 | 12·250 | 19 | 3 | 2 | 3 | 10 | 1·5809 |
| 0 | 16 | 11·625 | 19 | 3 | 1 | 3 | 10 | 0·2534 |
| 0 | 16 | 11·000 | 19 | 3 | 0 | 3 | 9 | 10·9260 |
| 0 | 16 | 10·375 | 19 | 2 | 7 | 3 | 9 | 9·5986 |
| 0 | 16 | 9·750 | 19 | 2 | 6 | 3 | 9 | 8·2712 |
| 0 | 16 | 9·125 | 19 | 2 | 5 | 3 | 9 | 6·9437 |
| 0 | 16 | 8·500 | 19 | 2 | 4 | 3 | 9 | 5·6163 |
| 0 | 16 | 7·875 | 19 | 2 | 3 | 3 | 9 | 4·2889 |
| 0 | 16 | 7·250 | 19 | 2 | 2 | 3 | 9 | 2·9615 |
| 0 | 16 | 6·625 | 19 | 2 | 1 | 3 | 9 | 1·6341 |
| 0 | 16 | 6·000 | 19 | 2 | 0 | 3 | 9 | 0·3067 |
| 0 | 16 | 5·375 | 19 | 1 | 7 | 3 | 8 | 10·9793 |
| 0 | 16 | 4·750 | 19 | 1 | 6 | 3 | 8 | 9·6518 |
| 0 | 16 | 4·125 | 19 | 1 | 5 | 3 | 8 | 8·3244 |
| 0 | 16 | 3·500 | 19 | 1 | 4 | 3 | 8 | 6·9970 |
| 0 | 16 | 2·875 | 19 | 1 | 3 | 3 | 8 | 5·6696 |
| 0 | 16 | 2·250 | 19 | 1 | 2 | 3 | 8 | 4·3422 |
| 0 | 16 | 1·625 | 19 | 1 | 1 | 3 | 8 | 3·0148 |
| 0 | 16 | 1·000 | 19 | 1 | 0 | 3 | 8 | 1·6874 |
| 0 | 16 | 0·375 | 19 | 0 | 7 | 3 | 8 | 0·3599 |
| 0 | 15 | 23·750 | 19 | 0 | 6 | 3 | 7 | 11·0325 |
| 0 | 15 | 23·125 | 19 | 0 | 5 | 3 | 7 | 9·7051 |
| 0 | 15 | 22·500 | 19 | 0 | 4 | 3 | 7 | 8·3777 |
| 0 | 15 | 21·875 | 19 | 0 | 3 | 3 | 7 | 7·0503 |
| 0 | 15 | 21·250 | 19 | 0 | 2 | 3 | 7 | 5·7229 |
| 0 | 15 | 20·625 | 19 | 0 | 1 | 3 | 7 | 4·3955 |
| 0 | 15 | 20·000 | 19 | 0 | 0 | 3 | 7 | 3·0681 |
| 0 | 15 | 19·375 | 18 | 3 | 7 | 3 | 7 | 1·7407 |
| 0 | 15 | 18·750 | 18 | 3 | 6 | 3 | 7 | 0·4133 |
| 0 | 15 | 18·125 | 18 | 3 | 5 | 3 | 6 | 11·0859 |
| 0 | 15 | 17·500 | 18 | 3 | 4 | 3 | 6 | 9·7585 |
| 0 | 15 | 16·875 | 18 | 3 | 3 | 3 | 6 | 8·4311 |
| 0 | 15 | 16·250 | 18 | 3 | 2 | 3 | 6 | 7·1036 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 15 | 15·625 | 18 | 3 | 1 | 3 | 6 | 5·7762 |
| 0 | 15 | 15·000 | 18 | 3 | 0 | 3 | 6 | 4·4488 |
| 0 | 15 | 14·375 | 18 | 2 | 7 | 3 | 6 | 3·1214 |
| 0 | 15 | 13·750 | 18 | 2 | 6 | 3 | 6 | 1·7940 |
| 0 | 15 | 13·125 | 18 | 2 | 5 | 3 | 6 | 0·4666 |
| 0 | 15 | 12·500 | 18 | 2 | 4 | 3 | 5 | 11·1392 |
| 0 | 15 | 11·875 | 18 | 2 | 3 | 3 | 5 | 9·8117 |
| 0 | 15 | 11·250 | 18 | 2 | 2 | 3 | 5 | 8·4843 |
| 0 | 15 | 10·625 | 18 | 2 | 1 | 3 | 5 | 7·1569 |
| 0 | 15 | 10·000 | 18 | 2 | 0 | 3 | 5 | 5·8295 |
| 0 | 15 | 9·375 | 18 | 1 | 7 | 3 | 5 | 4·5021 |
| 0 | 15 | 8·750 | 18 | 1 | 6 | 3 | 5 | 3·1747 |
| 0 | 15 | 8·125 | 18 | 1 | 5 | 3 | 5 | 1·8473 |
| 0 | 15 | 7·500 | 18 | 1 | 4 | 3 | 5 | 0·5198 |
| 0 | 15 | 6·875 | 18 | 1 | 3 | 3 | 4 | 11·1924 |
| 0 | 15 | 6·250 | 18 | 1 | 2 | 3 | 4 | 9·8650 |
| 0 | 15 | 5·625 | 18 | 1 | 1 | 3 | 4 | 8·5376 |
| 0 | 15 | 5·000 | 18 | 1 | 0 | 3 | 4 | 7·2102 |
| 0 | 15 | 4·375 | 18 | 0 | 7 | 3 | 4 | 5·8828 |
| 0 | 15 | 3·750 | 18 | 0 | 6 | 3 | 4 | 4·5554 |
| 0 | 15 | 3·125 | 18 | 0 | 5 | 3 | 4 | 3·2279 |
| 0 | 15 | 2·500 | 18 | 0 | 4 | 3 | 4 | 1·9005 |
| 0 | 15 | 1·875 | 18 | 0 | 3 | 3 | 4 | 0·5731 |
| 0 | 15 | 1·250 | 18 | 0 | 2 | 3 | 3 | 11·2457 |
| 0 | 15 | 0·625 | 18 | 0 | 1 | 3 | 3 | 9·9183 |
| 0 | 15 | 0·000 | 18 | 0 | 0 | 3 | 3 | 8·5909 |
| 0 | 14 | 23·375 | 17 | 3 | 7 | 3 | 3 | 7·2634 |
| 0 | 14 | 22·750 | 17 | 3 | 6 | 3 | 3 | 5·9360 |
| 0 | 14 | 22·125 | 17 | 3 | 5 | 3 | 3 | 4·6086 |
| 0 | 14 | 21·500 | 17 | 3 | 4 | 3 | 3 | 3·2812 |
| 0 | 14 | 20·875 | 17 | 3 | 3 | 3 | 3 | 1·9538 |
| 0 | 14 | 20·250 | 17 | 3 | 2 | 3 | 3 | 0·6264 |
| 0 | 14 | 19·625 | 17 | 3 | 1 | 3 | 2 | 11·2990 |
| 0 | 14 | 19·000 | 17 | 3 | 0 | 3 | 2 | 9·9715 |
| 0 | 14 | 18·375 | 17 | 2 | 7 | 3 | 2 | 8·6441 |
| 0 | 14 | 17·750 | 17 | 2 | 6 | 3 | 2 | 7·3167 |
| 0 | 14 | 17·125 | 17 | 2 | 5 | 3 | 2 | 5·9893 |
| 0 | 14 | 16·500 | 17 | 2 | 4 | 3 | 2 | 4·6619 |
| 0 | 14 | 15·875 | 17 | 2 | 3 | 3 | 2 | 3·3345 |
| 0 | 14 | 15·250 | 17 | 2 | 2 | 3 | 2 | 2·0071 |
| 0 | 14 | 14·625 | 17 | 2 | 1 | 3 | 2 | 0·6796 |
| 0 | 14 | 14·000 | 17 | 2 | 0 | 3 | 1 | 11·3522 |
| 0 | 14 | 13·375 | 17 | 1 | 7 | 3 | 1 | 10·0248 |
| 0 | 14 | 12·750 | 17 | 1 | 6 | 3 | 1 | 8·6974 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 14 | 12·125 | 17 | 1 | 5 | 3 | 1 | 7·3700 |
| 0 | 14 | 11·500 | 17 | 1 | 4 | 3 | 1 | 6·0426 |
| 0 | 14 | 10·875 | 17 | 1 | 3 | 3 | 1 | 4·7152 |
| 0 | 14 | 10·250 | 17 | 1 | 2 | 3 | 1 | 3·3877 |
| 0 | 14 | 9·625 | 17 | 1 | 1 | 3 | 1 | 2·0603 |
| 0 | 14 | 9·000 | 17 | 1 | 0 | 3 | 1 | 0·7329 |
| 0 | 14 | 8·375 | 17 | 0 | 7 | 3 | 0 | 11·4055 |
| 0 | 14 | 7·750 | 17 | 0 | 6 | 3 | 0 | 10·0781 |
| 0 | 14 | 7·125 | 17 | 0 | 5 | 3 | 0 | 8·7507 |
| 0 | 14 | 6·500 | 17 | 0 | 4 | 3 | 0 | 7·4233 |
| 0 | 14 | 5·875 | 17 | 0 | 3 | 3 | 0 | 6·0958 |
| 0 | 14 | 5·250 | 17 | 0 | 2 | 3 | 0 | 4·7684 |
| 0 | 14 | 4·625 | 17 | 0 | 1 | 3 | 0 | 3·4410 |
| 0 | 14 | 4·000 | 17 | 0 | 0 | 3 | 0 | 2·1136 |
| 0 | 14 | 3·375 | 16 | 3 | 7 | 3 | 0 | 0·7862 |
| 0 | 14 | 2·750 | 16 | 3 | 6 | 2 | 19 | 11·4588 |
| 0 | 14 | 2·125 | 16 | 3 | 5 | 2 | 19 | 10·1313 |
| 0 | 14 | 1·500 | 16 | 3 | 4 | 2 | 19 | 8·8039 |
| 0 | 14 | 0·875 | 16 | 3 | 3 | 2 | 19 | 7·4765 |
| 0 | 14 | 0·250 | 16 | 3 | 2 | 2 | 19 | 6·1491 |
| 0 | 13 | 23·625 | 16 | 3 | 1 | 2 | 19 | 4·8217 |
| 0 | 13 | 23·000 | 16 | 3 | 0 | 2 | 19 | 3·4943 |
| 0 | 13 | 22·375 | 16 | 2 | 7 | 2 | 19 | 2·1669 |
| 0 | 13 | 21·750 | 16 | 2 | 6 | 2 | 19 | 0·8394 |
| 0 | 13 | 21·125 | 16 | 2 | 5 | 2 | 18 | 11·5120 |
| 0 | 13 | 20·500 | 16 | 2 | 4 | 2 | 18 | 10·1846 |
| 0 | 13 | 19·875 | 16 | 2 | 3 | 2 | 18 | 8·8572 |
| 0 | 13 | 19·250 | 16 | 2 | 2 | 2 | 18 | 7·5298 |
| 0 | 13 | 18·625 | 16 | 2 | 1 | 2 | 18 | 6·2024 |
| 0 | 13 | 18·000 | 16 | 2 | 0 | 2 | 18 | 4·8750 |
| 0 | 13 | 17·375 | 16 | 1 | 7 | 2 | 18 | 3·5475 |
| 0 | 13 | 16·750 | 16 | 1 | 6 | 2 | 18 | 2·2201 |
| 0 | 13 | 16·125 | 16 | 1 | 5 | 2 | 18 | 0·8927 |
| 0 | 13 | 15·500 | 16 | 1 | 4 | 2 | 17 | 11·5653 |
| 0 | 13 | 14·875 | 16 | 1 | 3 | 2 | 17 | 10·2377 |
| 0 | 13 | 14·250 | 16 | 1 | 2 | 2 | 17 | 8·9103 |
| 0 | 13 | 13·625 | 16 | 1 | 1 | 2 | 17 | 7·5829 |
| 0 | 13 | 13·000 | 16 | 1 | 0 | 2 | 17 | 6·2554 |
| 0 | 13 | 12·375 | 16 | 0 | 7 | 2 | 17 | 4·9280 |
| 0 | 13 | 11·750 | 16 | 0 | 6 | 2 | 17 | 3·6006 |
| 0 | 13 | 11·125 | 16 | 0 | 5 | 2 | 17 | 2·2732 |
| 0 | 13 | 10·500 | 16 | 0 | 4 | 2 | 17 | 0·9458 |
| 0 | 13 | 9·875 | 16 | 0 | 3 | 2 | 16 | 11·6184 |
| 0 | 13 | 9·250 | 16 | 0 | 2 | 2 | 16 | 10·2909 |

| **Fine Gold,** Per Ounce | | | **Carat Gold,** Per Ounce | | | **Sterling Value,** Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 13 | 8·625 | 16 | 0 | 1 | 2 | 16 | 8·9635 |
| 0 | 13 | 8·000 | 16 | 0 | 0 | 2 | 16 | 7·6363 |
| 0 | 13 | 7·375 | 15 | 3 | 7 | 2 | 16 | 6·3089 |
| 0 | 13 | 6·750 | 15 | 3 | 6 | 2 | 16 | 4·9815 |
| 0 | 13 | 6·125 | 15 | 3 | 5 | 2 | 16 | 3·6541 |
| 0 | 13 | 5·500 | 15 | 3 | 4 | 2 | 16 | 2·3267 |
| 0 | 13 | 4.875 | 15 | 3 | 3 | 2 | 16 | 0·9992 |
| 0 | 13 | 4·250 | 15 | 3 | 2 | 2 | 15 | 11·6718 |
| 0 | 13 | 3·625 | 15 | 3 | 1 | 2 | 15 | 10·3444 |
| 0 | 13 | 3·000 | 15 | 3 | 0 | 2 | 15 | 9·0170 |
| 0 | 13 | 2·373 | 15 | 2 | 7 | 2 | 15 | 7·6896 |
| 0 | 13 | 1·750 | 15 | 2 | 6 | 2 | 15 | 6·3622 |
| 0 | 13 | 1·125 | 15 | 2 | 5 | 2 | 15 | 5·0348 |
| 0 | 13 | 0·500 | 15 | 2 | 4 | 2 | 15 | 3·7073 |
| 0 | 12 | 23·875 | 15 | 2 | 3 | 2 | 15 | 2·3799 |
| 0 | 12 | 23·250 | 15 | 2 | 2 | 2 | 15 | 1·0525 |
| 0 | 12 | 22·625 | 15 | 2 | 1 | 2 | 14 | 11·7251 |
| 0 | 12 | 22·000 | 15 | 2 | 0 | 2 | 14 | 10·3976 |
| 0 | 12 | 21·375 | 15 | 1 | 7 | 2 | 14 | 9·0702 |
| 0 | 12 | 20·750 | 15 | 1 | 6 | 2 | 14 | 7·7428 |
| 0 | 12 | 20·125 | 15 | 1 | 5 | 2 | 14 | 6·4154 |
| 0 | 12 | 19·500 | 15 | 1 | 4 | 2 | 14 | 5·0880 |
| 0 | 12 | 18·875 | 15 | 1 | 3 | 2 | 14 | 3·7606 |
| 0 | 12 | 18·250 | 15 | 1 | 2 | 2 | 14 | 2·4332 |
| 0 | 12 | 17·625 | 15 | 1 | 1 | 2 | 14 | 1·1057 |
| 0 | 12 | 17·000 | 15 | 1 | 0 | 2 | 13 | 11·7783 |
| 0 | 12 | 16·375 | 15 | 0 | 7 | 2 | 13 | 10·4509 |
| 0 | 12 | 15·750 | 15 | 0 | 6 | 2 | 13 | 9·1235 |
| 0 | 12 | 15·125 | 15 | 0 | 5 | 2 | 13 | 7·7961 |
| 0 | 12 | 14·500 | 15 | 0 | 4 | 2 | 13 | 6·4687 |
| 0 | 12 | 13·875 | 15 | 0 | 3 | 2 | 13 | 5·1413 |
| 0 | 12 | 13·250 | 15 | 0 | 2 | 2 | 13 | 3·8138 |
| 0 | 12 | 12·625 | 15 | 0 | 1 | 2 | 13 | 2·4864 |
| 0 | 12 | 12·000 | 15 | 0 | 0 | 2 | 13 | 1·1591 |
| 0 | 12 | 11·375 | 14 | 3 | 7 | 2 | 12 | 11·8316 |
| 0 | 12 | 10·750 | 14 | 3 | 6 | 2 | 12 | 10·5042 |
| 0 | 12 | 10·125 | 14 | 3 | 5 | 2 | 12 | 9·1768 |
| 0 | 12 | 9·500 | 14 | 3 | 4 | 2 | 12 | 7·8494 |
| 0 | 12 | 8·875 | 14 | 3 | 3 | 2 | 12 | 6·5220 |
| 0 | 12 | 8·250 | 14 | 3 | 2 | 2 | 12 | 5·1946 |
| 0 | 12 | 7·625 | 14 | 3 | 1 | 2 | 12 | 3·8671 |
| 0 | 12 | 7·000 | 14 | 3 | 0 | 2 | 12 | 2·5397 |
| 0 | 12 | 6·375 | 14 | 2 | 7 | 2 | 12 | 1·2123 |
| 0 | 12 | 5·750 | 14 | 2 | 6 | 2 | 11 | 11·8849 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 12 | 5·125 | 14 | 2 | 5 | 2 | 11 | 10·5575 |
| 0 | 12 | 4·500 | 14 | 2 | 4 | 2 | 11 | 9·2301 |
| 0 | 12 | 3·875 | 14 | 2 | 3 | 2 | 11 | 7·9027 |
| 0 | 12 | 3·250 | 14 | 2 | 2 | 2 | 11 | 6·5752 |
| 0 | 12 | 2·625 | 14 | 2 | 1 | 2 | 11 | 5·2478 |
| 0 | 12 | 2·000 | 14 | 2 | 0 | 2 | 11 | 3·9204 |
| 0 | 12 | 1·375 | 14 | 1 | 7 | 2 | 11 | 2·5930 |
| 0 | 12 | 0·750 | 14 | 1 | 6 | 2 | 11 | 1·2656 |
| 0 | 12 | 0·125 | 14 | 1 | 5 | 2 | 10 | 11·9382 |
| 0 | 11 | 23·500 | 14 | 1 | 4 | 2 | 10 | 10·6107 |
| 0 | 11 | 22·875 | 14 | 1 | 3 | 2 | 10 | 9·2833 |
| 0 | 11 | 22·250 | 14 | 1 | 2 | 2 | 10 | 7·9559 |
| 0 | 11 | 21·625 | 14 | 1 | 1 | 2 | 10 | 6·6285 |
| 0 | 11 | 21·000 | 14 | 1 | 0 | 2 | 10 | 5·3011 |
| 0 | 11 | 20·375 | 14 | 0 | 7 | 2 | 10 | 3·9737 |
| 0 | 11 | 19·750 | 14 | 0 | 6 | 2 | 10 | 2·6463 |
| 0 | 11 | 19·125 | 14 | 0 | 5 | 2 | 10 | 1·3188 |
| 0 | 11 | 18·500 | 14 | 0 | 4 | 2 | 9 | 11·9914 |
| 0 | 11 | 17·875 | 14 | 0 | 3 | 2 | 9 | 10·6640 |
| 0 | 11 | 17·250 | 14 | 0 | 2 | 2 | 9 | 9·3366 |
| 0 | 11 | 16·625 | 14 | 0 | 1 | 2 | 9 | 8·0092 |
| 0 | 11 | 16·000 | 14 | 0 | 0 | 2 | 9 | 6·6818 |
| 0 | 11 | 15·375 | 13 | 3 | 7 | 2 | 9 | 5·3544 |
| 0 | 11 | 14·750 | 13 | 3 | 6 | 2 | 9 | 4·0269 |
| 0 | 11 | 14·150 | 13 | 3 | 5 | 2 | 9 | 2·6995 |
| 0 | 11 | 13·500 | 13 | 3 | 4 | 2 | 9 | 1·3721 |
| 0 | 11 | 12·875 | 13 | 3 | 3 | 2 | 9 | 0·0447 |
| 0 | 11 | 12·250 | 13 | 3 | 2 | 2 | 8 | 10·7173 |
| 0 | 11 | 11·625 | 13 | 3 | 1 | 2 | 8 | 9·3899 |
| 0 | 11 | 11·000 | 13 | 3 | 0 | 2 | 8 | 8·0625 |
| 0 | 11 | 10·375 | 13 | 2 | 7 | 2 | 8 | 6·7350 |
| 0 | 11 | 9·750 | 13 | 2 | 6 | 2 | 8 | 5·4076 |
| 0 | 11 | 9·125 | 13 | 2 | 5 | 2 | 8 | 4·0802 |
| 0 | 11 | 8·500 | 13 | 2 | 4 | 2 | 8 | 2·7528 |
| 0 | 11 | 7·875 | 13 | 2 | 3 | 2 | 8 | 1·4254 |
| 0 | 11 | 7·250 | 13 | 2 | 2 | 2 | 8 | 0·0980 |
| 0 | 11 | 6·625 | 13 | 2 | 1 | 2 | 7 | 10·7705 |
| 0 | 11 | 6·000 | 13 | 2 | 0 | 2 | 7 | 9·4431 |
| 0 | 11 | 5·375 | 13 | 1 | 7 | 2 | 7 | 8·1157 |
| 0 | 11 | 4·750 | 13 | 1 | 6 | 2 | 7 | 6·7883 |
| 0 | 11 | 4·125 | 13 | 1 | 5 | 2 | 7 | 5·4609 |
| 0 | 11 | 3·500 | 13 | 1 | 4 | 2 | 7 | 4·1335 |
| 0 | 11 | 2·875 | 13 | 1 | 3 | 2 | 7 | 2·8061 |
| 0 | 11 | 2·250 | 13 | 1 | 2 | 2 | 7 | 1·4786 |

| **Fine Gold,** Per Ounce. | | | **Carat Gold,** Per Ounce | | | **Sterling Value,** Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 11 | 1·625 | 13 | 1 | 1 | 2 | 7 | 0·1512 |
| 0 | 11 | 1·000 | 13 | 1 | 0 | 2 | 6 | 10·8238 |
| 0 | 11 | 0·375 | 13 | 0 | 7 | 2 | 6 | 9·4964 |
| 0 | 10 | 23·750 | 13 | 0 | 6 | 2 | 6 | 8·1698 |
| 0 | 10 | 23·125 | 13 | 0 | 5 | 2 | 6 | 6·8416 |
| 0 | 10 | 22·500 | 13 | 0 | 4 | 2 | 6 | 5·5142 |
| 0 | 10 | 21·875 | 13 | 0 | 3 | 2 | 6 | 4·1867 |
| 0 | 10 | 21·250 | 13 | 0 | 2 | 2 | 6 | 2·8593 |
| 0 | 10 | 20·625 | 13 | 0 | 1 | 2 | 6 | 1·5319 |
| 0 | 10 | 20·000 | 13 | 0 | 0 | 2 | 6 | 0·2045 |
| 0 | 10 | 19·375 | 12 | 3 | 7 | 2 | 5 | 10·8771 |
| 0 | 10 | 18·750 | 12 | 3 | 6 | 2 | 5 | 9·5497 |
| 0 | 10 | 18·125 | 12 | 3 | 5 | 2 | 5 | 8·2223 |
| 0 | 10 | 17·500 | 12 | 3 | 4 | 2 | 5 | 6·8948 |
| 0 | 10 | 16·875 | 12 | 3 | 3 | 2 | 5 | 5·5674 |
| 0 | 10 | 16·250 | 12 | 3 | 2 | 2 | 5 | 4·2400 |
| 0 | 10 | 15·625 | 12 | 3 | 1 | 2 | 5 | 2·9126 |
| 0 | 10 | 15·000 | 12 | 3 | 0 | 2 | 5 | 1·5852 |
| 0 | 10 | 14·375 | 12 | 2 | 7 | 2 | 5 | 0·2578 |
| 0 | 10 | 13·750 | 12 | 2 | 6 | 2 | 4 | 10·9303 |
| 0 | 10 | 13·125 | 12 | 2 | 5 | 2 | 4 | 9·6029 |
| 0 | 10 | 12·500 | 12 | 2 | 4 | 2 | 4 | 8·2755 |
| 0 | 10 | 11·875 | 12 | 2 | 3 | 2 | 4 | 6·9481 |
| 0 | 10 | 11·250 | 12 | 2 | 2 | 2 | 4 | 5·6207 |
| 0 | 10 | 10·625 | 12 | 2 | 1 | 2 | 4 | 4·2933 |
| 0 | 10 | 10·000 | 12 | 2 | 0 | 2 | 4 | 2·9659 |
| 0 | 10 | 9·375 | 12 | 1 | 7 | 2 | 4 | 1·6384 |
| 0 | 10 | 8·750 | 12 | 1 | 6 | 2 | 4 | 0·3110 |
| 0 | 10 | 8·125 | 12 | 1 | 5 | 2 | 3 | 10·8366 |
| 0 | 10 | 7·500 | 12 | 1 | 4 | 2 | 3 | 9·6562 |
| 0 | 10 | 6·875 | 12 | 1 | 3 | 2 | 3 | 8·3288 |
| 0 | 10 | 6·250 | 12 | 1 | 2 | 2 | 3 | 7·0014 |
| 0 | 10 | 5·625 | 12 | 1 | 1 | 2 | 3 | 5·6740 |
| 0 | 10 | 5·000 | 12 | 1 | 0 | 2 | 3 | 4·3465 |
| 0 | 10 | 4·375 | 12 | 0 | 7 | 2 | 3 | 3·0191 |
| 0 | 10 | 3·750 | 12 | 0 | 6 | 2 | 3 | 1·6917 |
| 0 | 10 | 3·125 | 12 | 0 | 5 | 2 | 3 | 0·3643 |
| 0 | 10 | 2·500 | 12 | 0 | 4 | 2 | 2 | 11·0369 |
| 0 | 10 | 1·875 | 12 | 0 | 3 | 2 | 2 | 9·7095 |
| 0 | 10 | 1·250 | 12 | 0 | 2 | 2 | 2 | 8·3821 |
| 0 | 10 | 0·625 | 12 | 0 | 1 | 2 | 2 | 7·0546 |
| 0 | 10 | 0·000 | 12 | 0 | 0 | 2 | 2 | 5·7272 |
| 0 | 9 | 23·375 | 11 | 3 | 7 | 2 | 2 | 4·3998 |
| 0 | 9 | 22·750 | 11 | 3 | 6 | 2 | 2 | 3·0724 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 9 | 22·125 | 11 | 3 | 5 | 2 | 2 | 1·7450 |
| 0 | 9 | 21·500 | 11 | 3 | 4 | 2 | 2 | 0·4176 |
| 0 | 9 | 21·875 | 11 | 3 | 3 | 2 | 1 | 11·0901 |
| 0 | 9 | 20·250 | 11 | 3 | 2 | 2 | 1 | 9·7627 |
| 0 | 9 | 19·625 | 11 | 3 | 1 | 2 | 1 | 8·4353 |
| 0 | 9 | 19·000 | 11 | 3 | 0 | 2 | 1 | 7·1079 |
| 0 | 9 | 18·375 | 11 | 2 | 7 | 2 | 1 | 5·7805 |
| 0 | 9 | 17·750 | 11 | 2 | 6 | 2 | 1 | 4·4531 |
| 0 | 9 | 17·125 | 11 | 2 | 5 | 2 | 1 | 3·1257 |
| 0 | 9 | 16·500 | 11 | 2 | 4 | 2 | 1 | 1·7982 |
| 0 | 9 | 15·875 | 11 | 2 | 3 | 2 | 1 | 0·4708 |
| 0 | 9 | 15·250 | 11 | 2 | 2 | 2 | 0 | 11·1434 |
| 0 | 9 | 14·625 | 11 | 2 | 1 | 2 | 0 | 9·8160 |
| 0 | 9 | 14·000 | 11 | 2 | 0 | 2 | 0 | 8·4886 |
| 0 | 9 | 13·375 | 11 | 1 | 7 | 2 | 0 | 7·1612 |
| 0 | 9 | 12·750 | 11 | 1 | 6 | 2 | 0 | 5·8338 |
| 0 | 9 | 12·125 | 11 | 1 | 5 | 2 | 0 | 4·5063 |
| 0 | 9 | 11·500 | 11 | 1 | 4 | 2 | 0 | 3·1789 |
| 0 | 9 | 10·875 | 11 | 1 | 3 | 2 | 0 | 1·8515 |
| 0 | 9 | 10·250 | 11 | 1 | 2 | 2 | 0 | 0·5241 |
| 0 | 9 | 9·625 | 11 | 1 | 1 | 1 | 19 | 11·1967 |
| 0 | 9 | 9·000 | 11 | 1 | 0 | 1 | 19 | 9·8693 |
| 0 | 9 | 8·375 | 11 | 0 | 7 | 1 | 19 | 8·5419 |
| 0 | 9 | 7·750 | 11 | 0 | 6 | 1 | 19 | 7·2144 |
| 0 | 9 | 7·125 | 11 | 0 | 5 | 1 | 19 | 5·8870 |
| 0 | 9 | 6·500 | 11 | 0 | 4 | 1 | 19 | 4·5596 |
| 0 | 9 | 5·875 | 11 | 0 | 3 | 1 | 19 | 3·2322 |
| 0 | 9 | 5·250 | 11 | 0 | 2 | 1 | 19 | 1·9048 |
| 0 | 9 | 4·625 | 11 | 0 | 1 | 1 | 19 | 0·5774 |
| 0 | 9 | 4·000 | 11 | 0 | 0 | 1 | 18 | 11·2500 |
| 0 | 9 | 3·375 | 10 | 3 | 7 | 1 | 18 | 9·9225 |
| 0 | 9 | 2·750 | 10 | 3 | 6 | 1 | 18 | 8·5951 |
| 0 | 9 | 2·125 | 10 | 3 | 5 | 1 | 18 | 7·2677 |
| 0 | 9 | 1·500 | 10 | 3 | 4 | 1 | 18 | 5·9403 |
| 0 | 9 | 0·875 | 10 | 3 | 3 | 1 | 18 | 4·6129 |
| 0 | 9 | 0·250 | 10 | 3 | 2 | 1 | 18 | 3·2855 |
| 0 | 8 | 23·625 | 10 | 3 | 1 | 1 | 18 | 1·9580 |
| 0 | 8 | 23·000 | 10 | 3 | 0 | 1 | 18 | 0·6306 |
| 0 | 8 | 22·375 | 10 | 2 | 7 | 1 | 17 | 11·3032 |
| 0 | 8 | 21·750 | 10 | 2 | 6 | 1 | 17 | 9·9758 |
| 0 | 8 | 21·125 | 10 | 2 | 5 | 1 | 17 | 8·6484 |
| 0 | 8 | 20·500 | 10 | 2 | 4 | 1 | 17 | 7·3210 |
| 0 | 8 | 19·875 | 10 | 2 | 3 | 1 | 17 | 5·9936 |
| 0 | 8 | 19·250 | 10 | 2 | 2 | 1 | 17 | 4·6661 |

| Fine Gold,<br>Per Ounce | | | Carat Gold,<br>Per Ounce | | | Sterling Value,<br>Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 8 | 18·625 | 10 | 2 | 1 | 1 | 17 | 3·3387 |
| 0 | 8 | 18·000 | 10 | 2 | 0 | 1 | 17 | 2·0113 |
| 0 | 8 | 17·375 | 10 | 1 | 7 | 1 | 17 | 0·6839 |
| 0 | 8 | 16·750 | 10 | 1 | 6 | 1 | 16 | 11·3565 |
| 0 | 8 | 16·125 | 10 | 1 | 5 | 1 | 16 | 10·0291 |
| 0 | 8 | 15·500 | 10 | 1 | 4 | 1 | 16 | 8·7017 |
| 0 | 8 | 14·875 | 10 | 1 | 3 | 1 | 16 | 7·3742 |
| 0 | 8 | 14·250 | 10 | 1 | 2 | 1 | 16 | 6·0468 |
| 0 | 8 | 13·625 | 10 | 1 | 1 | 1 | 16 | 4·7194 |
| 0 | 8 | 13·000 | 10 | 1 | 0 | 1 | 16 | 3·3920 |
| 0 | 8 | 12·375 | 10 | 0 | 7 | 1 | 16 | 2·0646 |
| 0 | 8 | 11·750 | 10 | 0 | 6 | 1 | 16 | 0·7372 |
| 0 | 8 | 11·125 | 10 | 0 | 5 | 1 | 15 | 11·4098 |
| 0 | 8 | 10·500 | 10 | 0 | 4 | 1 | 15 | 10·0823 |
| 0 | 8 | 9·875 | 10 | 0 | 3 | 1 | 15 | 8·7549 |
| 0 | 8 | 9·250 | 10 | 0 | 2 | 1 | 15 | 7·4275 |
| 0 | 8 | 8·625 | 10 | 0 | 1 | 1 | 15 | 6·1001 |
| 0 | 8 | 8·000 | 10 | 0 | 0 | 1 | 15 | 4·7728 |
| 0 | 8 | 7·375 | 9 | 3 | 7 | 1 | 15 | 3·4454 |
| 0 | 8 | 6·750 | 9 | 3 | 6 | 1 | 15 | 2·1179 |
| 0 | 8 | 6·125 | 9 | 3 | 5 | 1 | 15 | 0·7905 |
| 0 | 8 | 5·500 | 9 | 3 | 4 | 1 | 14 | 11·4631 |
| 0 | 8 | 4·875 | 9 | 3 | 3 | 1 | 14 | 10·1357 |
| 0 | 8 | 4·250 | 9 | 3 | 2 | 1 | 14 | 8·8083 |
| 0 | 8 | 3·625 | 9 | 3 | 1 | 1 | 14 | 7·4809 |
| 0 | 8 | 3·000 | 9 | 3 | 0 | 1 | 14 | 6·1535 |
| 0 | 8 | 2·375 | 9 | 2 | 7 | 1 | 14 | 4·8260 |
| 0 | 8 | 1·750 | 9 | 2 | 6 | 1 | 14 | 3·4986 |
| 0 | 8 | 1·125 | 9 | 2 | 5 | 1 | 14 | 2·1712 |
| 0 | 8 | 0·500 | 9 | 2 | 4 | 1 | 14 | 0·8438 |
| 0 | 7 | 23·875 | 9 | 2 | 3 | 1 | 13 | 11·5164 |
| 0 | 7 | 23·250 | 9 | 2 | 2 | 1 | 13 | 10·1890 |
| 0 | 7 | 22·625 | 9 | 2 | 1 | 1 | 13 | 8·8616 |
| 0 | 7 | 22·000 | 9 | 2 | 0 | 1 | 13 | 7·5341 |
| 0 | 7 | 21·375 | 9 | 1 | 7 | 1 | 13 | 6·2067 |
| 0 | 7 | 20·750 | 9 | 1 | 6 | 1 | 13 | 4·8793 |
| 0 | 7 | 20·125 | 9 | 1 | 5 | 1 | 13 | 3·5519 |
| 0 | 7 | 19·500 | 9 | 1 | 4 | 1 | 13 | 2·2245 |
| 0 | 7 | 19·875 | 9 | 1 | 3 | 1 | 13 | 0·8971 |
| 0 | 7 | 18·250 | 9 | 1 | 2 | 1 | 12 | 11·5697 |
| 0 | 7 | 17·625 | 9 | 1 | 1 | 1 | 12 | 10·2422 |
| 0 | 7 | 17·000 | 9 | 1 | 0 | 1 | 12 | 8·9168 |
| 0 | 7 | 16·375 | 9 | 0 | 7 | 1 | 12 | 7·5874 |
| 0 | 7 | 15·750 | 9 | 0 | 6 | 1 | 12 | 6·2600 |

| FINE GOLD, Per Ounce | | | CARAT GOLD, Per Ounce | | | STERLING VALUE, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 7 | 15·125 | 9 | 0 | 5 | 1 | 12 | 4·9326 |
| 0 | 7 | 14·500 | 9 | 0 | 4 | 1 | 12 | 3·6052 |
| 0 | 7 | 13·875 | 9 | 0 | 3 | 1 | 12 | 2·2778 |
| 0 | 7 | 13·250 | 9 | 0 | 2 | 1 | 12 | 0·9503 |
| 0 | 7 | 12·625 | 9 | 0 | 1 | 1 | 11 | 11·6229 |
| 0 | 7 | 12·000 | 9 | 0 | 0 | 1 | 11 | 10·2954 |
| 0 | 7 | 11·375 | 8 | 3 | 7 | 1 | 11 | 8·9680 |
| 0 | 7 | 10·750 | 8 | 3 | 6 | 1 | 11 | 7·6406 |
| 0 | 7 | 10·125 | 8 | 3 | 5 | 1 | 11 | 6·3132 |
| 0 | 7 | 9·500 | 8 | 3 | 4 | 1 | 11 | 4·9857 |
| 0 | 7 | 8·875 | 8 | 3 | 3 | 1 | 11 | 3·6583 |
| 0 | 7 | 8·250 | 8 | 3 | 2 | 1 | 11 | 2·3309 |
| 0 | 7 | 7·625 | 8 | 3 | 1 | 1 | 11 | 1·0035 |
| 0 | 7 | 7·000 | 8 | 3 | 0 | 1 | 10 | 11·6761 |
| 0 | 7 | 6·375 | 8 | 2 | 7 | 1 | 10 | 10·3487 |
| 0 | 7 | 5·750 | 8 | 2 | 6 | 1 | 10 | 9·0213 |
| 0 | 7 | 5·125 | 8 | 2 | 5 | 1 | 10 | 7·6938 |
| 0 | 7 | 4·500 | 8 | 2 | 4 | 1 | 10 | 6·3664 |
| 0 | 7 | 3·875 | 8 | 2 | 3 | 1 | 10 | 5·0390 |
| 0 | 7 | 3·250 | 8 | 2 | 2 | 1 | 10 | 3·7116 |
| 0 | 7 | 2·625 | 8 | 2 | 1 | 1 | 10 | 2·3843 |
| 0 | 7 | 2·000 | 8 | 2 | 0 | 1 | 10 | 1·0568 |
| 0 | 7 | 1·375 | 8 | 1 | 7 | 1 | 9 | 11·7294 |
| 0 | 7 | 0·750 | 8 | 1 | 6 | 1 | 9 | 10·4019 |
| 0 | 7 | 0·125 | 8 | 1 | 5 | 1 | 9 | 9·0745 |
| 0 | 6 | 23·500 | 8 | 1 | 4 | 1 | 9 | 7·7471 |
| 0 | 6 | 22·875 | 8 | 1 | 3 | 1 | 9 | 6·4197 |
| 0 | 6 | 22·250 | 8 | 1 | 2 | 1 | 9 | 5·0923 |
| 0 | 6 | 21·625 | 8 | 1 | 1 | 1 | 9 | 3·7649 |
| 0 | 6 | 21·000 | 8 | 1 | 0 | 1 | 9 | 2·4375 |
| 0 | 6 | 20·375 | 8 | 0 | 7 | 1 | 9 | 1·1100 |
| 0 | 6 | 19·750 | 8 | 0 | 6 | 1 | 8 | 11·7826 |
| 0 | 6 | 19·125 | 8 | 0 | 5 | 1 | 8 | 10·4552 |
| 0 | 6 | 18·500 | 8 | 0 | 4 | 1 | 8 | 9·1278 |
| 0 | 6 | 17·875 | 8 | 0 | 3 | 1 | 8 | 7·8004 |
| 0 | 6 | 17·250 | 8 | 0 | 2 | 1 | 8 | 6·4730 |
| 0 | 6 | 16·625 | 8 | 0 | 1 | 1 | 8 | 5·1455 |
| 0 | 6 | 16·000 | 8 | 0 | 0 | 1 | 8 | 3·8181 |
| 0 | 6 | 15·375 | 7 | 3 | 7 | 1 | 8 | 2·4907 |
| 0 | 6 | 14·750 | 7 | 3 | 6 | 1 | 8 | 1·1633 |
| 0 | 6 | 14·125 | 7 | 3 | 5 | 1 | 7 | 11·8359 |
| 0 | 6 | 13·500 | 7 | 3 | 4 | 1 | 7 | 10·5085 |
| 0 | 6 | 12·875 | 7 | 3 | 3 | 1 | 7 | 9·1811 |
| 0 | 6 | 12·250 | 7 | 3 | 2 | 1 | 7 | 7·8536 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 6 | 11·625 | 7 | 3 | 1 | 1 | 7 | 6·5262 |
| 0 | 6 | 11·000 | 7 | 3 | 0 | 1 | 7 | 5·1988 |
| 0 | 6 | 10·375 | 7 | 2 | 7 | 1 | 7 | 3·8714 |
| 0 | 6 | 9·750 | 7 | 2 | 6 | 1 | 7 | 2·5440 |
| 0 | 6 | 9·125 | 7 | 2 | 5 | 1 | 7 | 1·2166 |
| 0 | 6 | 8·500 | 7 | 2 | 4 | 1 | 6 | 11·8892 |
| 0 | 6 | 7·875 | 7 | 2 | 3 | 1 | 6 | 10·5617 |
| 0 | 6 | 7·250 | 7 | 2 | 2 | 1 | 6 | 9·2343 |
| 0 | 6 | 6·625 | 7 | 2 | 1 | 1 | 6 | 7·8069 |
| 0 | 6 | 6·000 | 7 | 2 | 0 | 1 | 6 | 6·5795 |
| 0 | 6 | 5·375 | 7 | 1 | 7 | 1 | 6 | 5·2521 |
| 0 | 6 | 4·750 | 7 | 1 | 6 | 1 | 6 | 3·9247 |
| 0 | 6 | 4·125 | 7 | 1 | 5 | 1 | 6 | 2·5973 |
| 0 | 6 | 3·500 | 7 | 1 | 4 | 1 | 6 | 1·2698 |
| 0 | 6 | 2·875 | 7 | 1 | 3 | 1 | 5 | 11·9424 |
| 0 | 6 | 2·250 | 7 | 1 | 2 | 1 | 5 | 10·6150 |
| 0 | 6 | 1·625 | 7 | 1 | 1 | 1 | 5 | 9·2876 |
| 0 | 6 | 1·000 | 7 | 1 | 0 | 1 | 5 | 7·9602 |
| 0 | 6 | 0·375 | 7 | 0 | 7 | 1 | 5 | 6·6328 |
| 0 | 5 | 23·750 | 7 | 0 | 6 | 1 | 5 | 5·3054 |
| 0 | 5 | 23·125 | 7 | 0 | 5 | 1 | 5 | 3·9779 |
| 0 | 5 | 22·500 | 7 | 0 | 4 | 1 | 5 | 2·6505 |
| 0 | 5 | 21·875 | 7 | 0 | 3 | 1 | 5 | 1·3231 |
| 0 | 5 | 21·250 | 7 | 0 | 2 | 1 | 4 | 11·9957 |
| 0 | 5 | 20·625 | 7 | 0 | 1 | 1 | 4 | 10·6683 |
| 0 | 5 | 20·000 | 7 | 0 | 0 | 1 | 4 | 9·3409 |
| 0 | 5 | 19·375 | 6 | 3 | 7 | 1 | 4 | 8·0134 |
| 0 | 5 | 18·750 | 6 | 3 | 6 | 1 | 4 | 6·6860 |
| 0 | 5 | 18·125 | 6 | 3 | 5 | 1 | 4 | 5·3586 |
| 0 | 5 | 17·500 | 6 | 3 | 4 | 1 | 4 | 4·0312 |
| 0 | 5 | 16·875 | 6 | 3 | 3 | 1 | 4 | 2·7038 |
| 0 | 5 | 16·250 | 6 | 3 | 2 | 1 | 4 | 1·3764 |
| 0 | 5 | 15·625 | 6 | 3 | 1 | 1 | 4 | 0·0490 |
| 0 | 5 | 15·000 | 6 | 3 | 0 | 1 | 3 | 10·7216 |
| 0 | 5 | 14·375 | 6 | 2 | 7 | 1 | 3 | 9·3941 |
| 0 | 5 | 13·750 | 6 | 2 | 6 | 1 | 3 | 8·0667 |
| 0 | 5 | 13·125 | 6 | 2 | 5 | 1 | 3 | 6·7393 |
| 0 | 5 | 12·500 | 6 | 2 | 4 | 1 | 3 | 5·4119 |
| 0 | 5 | 11·875 | 6 | 2 | 3 | 1 | 3 | 4·0845 |
| 0 | 5 | 11·250 | 6 | 2 | 2 | 1 | 3 | 2·7571 |
| 0 | 5 | 10·625 | 6 | 2 | 1 | 1 | 3 | 1·4297 |
| 0 | 5 | 10·000 | 6 | 2 | 0 | 1 | 3 | 0·1022 |
| 0 | 5 | 9·375 | 6 | 1 | 7 | 1 | 2 | 10·7748 |
| 0 | 5 | 8·750 | 6 | 1 | 6 | 1 | 2 | 9·4474 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 5 | 8·125 | 6 | 1 | 5 | 1 | 2 | 8·1200 |
| 0 | 5 | 7·500 | 6 | 1 | 4 | 1 | 2 | 6·7926 |
| 0 | 5 | 6·875 | 6 | 1 | 3 | 1 | 2 | 5·4652 |
| 0 | 5 | 6·250 | 6 | 1 | 2 | 1 | 2 | 4·1377 |
| 0 | 5 | 5·625 | 6 | 1 | 1 | 1 | 2 | 2·8103 |
| 0 | 5 | 5·000 | 6 | 1 | 0 | 1 | 2 | 1·4829 |
| 0 | 5 | 4·375 | 6 | 0 | 7 | 1 | 2 | 0·1555 |
| 0 | 5 | 3·750 | 6 | 0 | 6 | 1 | 1 | 10·8281 |
| 0 | 5 | 3·125 | 6 | 0 | 5 | 1 | 1 | 9·5007 |
| 0 | 5 | 2·500 | 6 | 0 | 4 | 1 | 1 | 8·1733 |
| 0 | 5 | 1·875 | 6 | 0 | 3 | 1 | 1 | 6·8458 |
| 0 | 5 | 1·250 | 6 | 0 | 2 | 1 | 1 | 5·5184 |
| 0 | 5 | 0·625 | 6 | 0 | 1 | 1 | 1 | 4·1910 |
| 0 | 5 | 0·000 | 6 | 0 | 0 | 1 | 1 | 2·8636 |
| 0 | 4 | 23·375 | 5 | 3 | 7 | 1 | 1 | 1·5362 |
| 0 | 4 | 22·750 | 5 | 3 | 6 | 1 | 1 | 0·2088 |
| 0 | 4 | 22·125 | 5 | 3 | 5 | 1 | 0 | 10·8813 |
| 0 | 4 | 21·500 | 5 | 3 | 4 | 1 | 0 | 9·5539 |
| 0 | 4 | 20·875 | 5 | 3 | 3 | 1 | 0 | 8·2265 |
| 0 | 4 | 20·250 | 5 | 3 | 2 | 1 | 0 | 6·8991 |
| 0 | 4 | 19·625 | 5 | 3 | 1 | 1 | 0 | 5·5717 |
| 0 | 4 | 19·000 | 5 | 3 | 0 | 1 | 0 | 4·2443 |
| 0 | 4 | 18·375 | 5 | 2 | 7 | 1 | 0 | 2·9169 |
| 0 | 4 | 17·750 | 5 | 2 | 6 | 1 | 0 | 1·5894 |
| 0 | 4 | 17·125 | 5 | 2 | 5 | 1 | 0 | 0·2620 |
| 0 | 4 | 16·500 | 5 | 2 | 4 | 0 | 19 | 10·9346 |
| 0 | 4 | 15·875 | 5 | 2 | 3 | 0 | 19 | 9·6072 |
| 0 | 4 | 15·250 | 5 | 2 | 2 | 0 | 19 | 8·2798 |
| 0 | 4 | 14·625 | 5 | 2 | 1 | 0 | 19 | 6·9524 |
| 0 | 4 | 14·000 | 5 | 2 | 0 | 0 | 19 | 5·6250 |
| 0 | 4 | 13·375 | 5 | 1 | 7 | 0 | 19 | 4·2975 |
| 0 | 4 | 12·750 | 5 | 1 | 6 | 0 | 19 | 2·9701 |
| 0 | 4 | 12·125 | 5 | 1 | 5 | 0 | 19 | 1·6427 |
| 0 | 4 | 11·500 | 5 | 1 | 4 | 0 | 19 | 0·3153 |
| 0 | 4 | 10·875 | 5 | 1 | 3 | 0 | 18 | 10·9879 |
| 0 | 4 | 10·250 | 5 | 1 | 2 | 0 | 18 | 9·6605 |
| 0 | 4 | 9·625 | 5 | 1 | 1 | 0 | 18 | 8·3331 |
| 0 | 4 | 9·000 | 5 | 1 | 0 | 0 | 18 | 7·0056 |
| 0 | 4 | 8·375 | 5 | 0 | 7 | 0 | 18 | 5·6782 |
| 0 | 4 | 7·750 | 5 | 0 | 6 | 0 | 18 | 4·3508 |
| 0 | 4 | 7·125 | 5 | 0 | 5 | 0 | 18 | 3·0234 |
| 0 | 4 | 6·500 | 5 | 0 | 4 | 0 | 18 | 1·6960 |
| 0 | 4 | 5·875 | 5 | 0 | 3 | 0 | 18 | 0·3686 |
| 0 | 4 | 5·250 | 5 | 0 | 2 | 0 | 17 | 11·0411 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 4 | 4·625 | 5 | 0 | 1 | 0 | 17 | 9·7137 |
| 0 | 4 | 4·000 | 5 | 0 | 0 | 0 | 17 | 8·3863 |
| 0 | 4 | 3·375 | 4 | 3 | 7 | 0 | 17 | 7·0589 |
| 0 | 4 | 2·750 | 4 | 3 | 6 | 0 | 17 | 5·7315 |
| 0 | 4 | 2·125 | 4 | 3 | 5 | 0 | 17 | 4·4041 |
| 0 | 4 | 1·500 | 4 | 3 | 4 | 0 | 17 | 3·0767 |
| 0 | 4 | 0·875 | 4 | 3 | 3 | 0 | 17 | 1·7492 |
| 0 | 4 | 0·250 | 4 | 3 | 2 | 0 | 17 | 0·4218 |
| 0 | 3 | 23·625 | 4 | 3 | 1 | 0 | 16 | 11·0944 |
| 0 | 3 | 23·000 | 4 | 3 | 0 | 0 | 16 | 9·7670 |
| 0 | 3 | 22·375 | 4 | 2 | 7 | 0 | 16 | 8·4396 |
| 0 | 3 | 21·750 | 4 | 2 | 6 | 0 | 16 | 7·1122 |
| 0 | 3 | 21·125 | 4 | 2 | 5 | 0 | 16 | 5·7848 |
| 0 | 3 | 20·500 | 4 | 2 | 4 | 0 | 16 | 4·4573 |
| 0 | 3 | 19·875 | 4 | 2 | 3 | 0 | 16 | 3·1299 |
| 0 | 3 | 19·250 | 4 | 2 | 2 | 0 | 16 | 1·8025 |
| 0 | 3 | 18·625 | 4 | 2 | 1 | 0 | 16 | 0·4751 |
| 0 | 3 | 18·000 | 4 | 2 | 0 | 0 | 15 | 11·1477 |
| 0 | 3 | 17·375 | 4 | 1 | 7 | 0 | 15 | 9·8203 |
| 0 | 3 | 16·750 | 4 | 1 | 6 | 0 | 15 | 8·4929 |
| 0 | 3 | 16·125 | 4 | 1 | 5 | 0 | 15 | 7·1655 |
| 0 | 3 | 15·500 | 4 | 1 | 4 | 0 | 15 | 5·8380 |
| 0 | 3 | 14·875 | 4 | 1 | 3 | 0 | 15 | 4·5106 |
| 0 | 3 | 14·250 | 4 | 1 | 2 | 0 | 15 | 3·1832 |
| 0 | 3 | 13·625 | 4 | 1 | 1 | 0 | 15 | 1·8558 |
| 0 | 3 | 13·000 | 4 | 1 | 0 | 0 | 15 | 0·5284 |
| 0 | 3 | 12·375 | 4 | 0 | 7 | 0 | 14 | 11·2009 |
| 0 | 3 | 11·750 | 4 | 0 | 6 | 0 | 14 | 9·8735 |
| 0 | 3 | 11·125 | 4 | 0 | 5 | 0 | 14 | 8·5461 |
| 0 | 3 | 10·500 | 4 | 0 | 4 | 0 | 14 | 7·2187 |
| 0 | 3 | 9·875 | 4 | 0 | 3 | 0 | 14 | 5·8913 |
| 0 | 3 | 9·250 | 4 | 0 | 2 | 0 | 14 | 4·5639 |
| 0 | 3 | 8·625 | 4 | 0 | 1 | 0 | 14 | 3·2365 |
| 0 | 3 | 8·000 | 4 | 0 | 0 | 0 | 14 | 1·9090 |
| 0 | 3 | 7·375 | 3 | 3 | 7 | 0 | 14 | 0·5816 |
| 0 | 3 | 6·750 | 3 | 3 | 6 | 0 | 13 | 11·2542 |
| 0 | 3 | 6·125 | 3 | 3 | 5 | 0 | 13 | 9·9268 |
| 0 | 3 | 5·500 | 3 | 3 | 4 | 0 | 13 | 8·5994 |
| 0 | 3 | 4·875 | 3 | 3 | 3 | 0 | 13 | 7·2720 |
| 0 | 3 | 4·250 | 3 | 3 | 2 | 0 | 13 | 5·9446 |
| 0 | 3 | 3·625 | 3 | 3 | 1 | 0 | 13 | 4·6171 |
| 0 | 3 | 3·000 | 3 | 3 | 0 | 0 | 13 | 3·2897 |
| 0 | 3 | 2·375 | 3 | 2 | 7 | 0 | 13 | 1·9623 |
| 0 | 3 | 1·750 | 3 | 2 | 6 | 0 | 13 | 0·6349 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carat.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 3 | 1·125 | 3 | 2 | 5 | 0 | 12 | 11·3075 |
| 0 | 3 | 0·500 | 3 | 2 | 4 | 0 | 12 | 9·9801 |
| 0 | 2 | 23·875 | 3 | 2 | 3 | 0 | 12 | 8·6527 |
| 0 | 2 | 23·250 | 3 | 2 | 2 | 0 | 12 | 7·3250 |
| 0 | 2 | 22·625 | 3 | 2 | 1 | 0 | 12 | 5·9978 |
| 0 | 2 | 22·000 | 3 | 2 | 0 | 0 | 12 | 4·6704 |
| 0 | 2 | 21·375 | 3 | 1 | 7 | 0 | 12 | 3·3430 |
| 0 | 2 | 20·750 | 3 | 1 | 6 | 0 | 12 | 2·0156 |
| 0 | 2 | 20·125 | 3 | 1 | 5 | 0 | 12 | 0·6882 |
| 0 | 2 | 19·500 | 3 | 1 | 4 | 0 | 11 | 11·3607 |
| 0 | 2 | 18·875 | 3 | 1 | 3 | 0 | 11 | 10·0333 |
| 0 | 2 | 18·250 | 3 | 1 | 2 | 0 | 11 | 8·7059 |
| 0 | 2 | 17·625 | 3 | 1 | 1 | 0 | 11 | 7·3785 |
| 0 | 2 | 17·000 | 3 | 1 | 0 | 0 | 11 | 6·0511 |
| 0 | 2 | 16·375 | 3 | 0 | 7 | 0 | 11 | 4·7237 |
| 0 | 2 | 15·750 | 3 | 0 | 6 | 0 | 11 | 3·3963 |
| 0 | 2 | 15·125 | 3 | 0 | 5 | 0 | 11 | 2·0688 |
| 0 | 2 | 14·500 | 3 | 0 | 4 | 0 | 11 | 0·7414 |
| 0 | 2 | 13·875 | 3 | 0 | 3 | 0 | 10 | 11·4140 |
| 0 | 2 | 13·250 | 3 | 0 | 2 | 0 | 10 | 10·0866 |
| 0 | 2 | 12·625 | 3 | 0 | 1 | 0 | 10 | 8·7592 |
| 0 | 2 | 12·000 | 3 | 0 | 0 | 0 | 10 | 7·4318 |
| 0 | 2 | 11·375 | 2 | 3 | 7 | 0 | 10 | 6·1044 |
| 0 | 2 | 10·750 | 2 | 3 | 6 | 0 | 10 | 4·7769 |
| 0 | 2 | 10·125 | 2 | 3 | 5 | 0 | 10 | 3·4495 |
| 0 | 2 | 9·500 | 2 | 3 | 4 | 0 | 10 | 2·1221 |
| 0 | 2 | 8·875 | 2 | 3 | 3 | 0 | 10 | 0·7947 |
| 0 | 2 | 8·250 | 2 | 3 | 2 | 0 | 9 | 11·4673 |
| 0 | 2 | 7·625 | 2 | 3 | 1 | 0 | 9 | 10·1399 |
| 0 | 2 | 7·000 | 2 | 3 | 0 | 0 | 9 | 8·8125 |
| 0 | 2 | 6·375 | 2 | 2 | 7 | 0 | 9 | 7·4850 |
| 0 | 2 | 5·750 | 2 | 2 | 6 | 0 | 9 | 6·1576 |
| 0 | 2 | 5·125 | 2 | 2 | 5 | 0 | 9 | 4·8302 |
| 0 | 2 | 4·500 | 2 | 2 | 4 | 0 | 9 | 3·5028 |
| 0 | 2 | 3·875 | 2 | 2 | 3 | 0 | 9 | 2·1754 |
| 0 | 2 | 3·250 | 2 | 2 | 2 | 0 | 9 | 0·8480 |
| 0 | 2 | 2·625 | 2 | 2 | 1 | 0 | 8 | 11·5205 |
| 0 | 2 | 2·000 | 2 | 2 | 0 | 0 | 8 | 10·1931 |
| 0 | 2 | 1·375 | 2 | 1 | 7 | 0 | 8 | 8·8657 |
| 0 | 2 | 0·750 | 2 | 1 | 6 | 0 | 8 | 7·5383 |
| 0 | 2 | 0·125 | 2 | 1 | 5 | 0 | 8 | 6·2109 |
| 0 | 1 | 23·500 | 2 | 1 | 4 | 0 | 8 | 4·8835 |
| 0 | 1 | 22·875 | 2 | 1 | 3 | 0 | 8 | 3·5561 |
| 0 | 1 | 22·250 | 2 | 1 | 2 | 0 | 8 | 2·2286 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 1 | 21·625 | 2 | 1 | 1 | 0 | 8 | 0·9012 |
| 0 | 1 | 21·000 | 2 | 1 | 0 | 0 | 7 | 11·5738 |
| 0 | 1 | 20·375 | 2 | 0 | 7 | 0 | 7 | 10·2464 |
| 0 | 1 | 19·750 | 2 | 0 | 6 | 0 | 7 | 8·9190 |
| 0 | 1 | 19·125 | 2 | 0 | 5 | 0 | 7 | 7·5916 |
| 0 | 1 | 18·500 | 2 | 0 | 4 | 0 | 7 | 6·2642 |
| 0 | 1 | 17·875 | 2 | 0 | 3 | 0 | 7 | 4·9367 |
| 0 | 1 | 17·250 | 2 | 0 | 2 | 0 | 7 | 3·6093 |
| 0 | 1 | 16·625 | 2 | 0 | 1 | 0 | 7 | 2·2819 |
| 0 | 1 | 16·000 | 2 | 0 | 0 | 0 | 7 | 0·9545 |
| 0 | 1 | 15·375 | 1 | 3 | 7 | 0 | 6 | 11·6271 |
| 0 | 1 | 14·750 | 1 | 3 | 6 | 0 | 6 | 10·2997 |
| 0 | 1 | 14·125 | 1 | 3 | 5 | 0 | 6 | 8·9723 |
| 0 | 1 | 13·500 | 1 | 3 | 4 | 0 | 6 | 7·6448 |
| 0 | 1 | 12·875 | 1 | 3 | 3 | 0 | 6 | 6·3174 |
| 0 | 1 | 12·250 | 1 | 3 | 2 | 0 | 6 | 4·9900 |
| 0 | 1 | 11·625 | 1 | 3 | 1 | 0 | 6 | 3·6626 |
| 0 | 1 | 11·000 | 1 | 3 | 0 | 0 | 6 | 2·3352 |
| 0 | 1 | 10·375 | 1 | 2 | 7 | 0 | 6 | 1·0078 |
| 0 | 1 | 9·750 | 1 | 2 | 6 | 0 | 5 | 11·6803 |
| 0 | 1 | 9·125 | 1 | 2 | 5 | 0 | 5 | 10·3529 |
| 0 | 1 | 8·500 | 1 | 2 | 4 | 0 | 5 | 9·0255 |
| 0 | 1 | 7·875 | 1 | 2 | 3 | 0 | 5 | 7·6981 |
| 0 | 1 | 7·250 | 1 | 2 | 2 | 0 | 5 | 6·3707 |
| 0 | 1 | 6·625 | 1 | 2 | 1 | 0 | 5 | 5·0433 |
| 0 | 1 | 6·000 | 1 | 2 | 0 | 0 | 5 | 3·7159 |
| 0 | 1 | 5·375 | 1 | 1 | 7 | 0 | 5 | 2·3884 |
| 0 | 1 | 4·750 | 1 | 1 | 6 | 0 | 5 | 1·0610 |
| 0 | 1 | 4·125 | 1 | 1 | 5 | 0 | 4 | 11·7336 |
| 0 | 1 | 3·500 | 1 | 1 | 4 | 0 | 4 | 10·4062 |
| 0 | 1 | 2·875 | 1 | 1 | 3 | 0 | 4 | 9·0788 |
| 0 | 1 | 2·250 | 1 | 1 | 2 | 0 | 4 | 7·7514 |
| 0 | 1 | 1·625 | 1 | 1 | 1 | 0 | 4 | 6·4240 |
| 0 | 1 | 1·000 | 1 | 1 | 0 | 0 | 4 | 5·0965 |
| 0 | 1 | 0·375 | 1 | 0 | 7 | 0 | 4 | 3·7691 |
| 0 | 0 | 23·750 | 1 | 0 | 6 | 0 | 4 | 2·4417 |
| 0 | 0 | 23·125 | 1 | 0 | 5 | 0 | 4 | 1·1143 |
| 0 | 0 | 22·500 | 1 | 0 | 4 | 0 | 3 | 11·7869 |
| 0 | 0 | 21·875 | 1 | 0 | 3 | 0 | 3 | 10·4595 |
| 0 | 0 | 21·250 | 1 | 0 | 2 | 0 | 3 | 9·1321 |
| 0 | 0 | 20·625 | 1 | 0 | 1 | 0 | 3 | 7·8046 |
| 0 | 0 | 20·000 | 1 | 0 | 0 | 0 | 3 | 6·4772 |
| 0 | 0 | 19·375 | 0 | 3 | 7 | 0 | 3 | 5·1498 |
| 0 | 0 | 18·750 | 0 | 3 | 6 | 0 | 3 | 3·8224 |

| Fine Gold, Per Ounce | | | Carat Gold, Per Ounce | | | Sterling Value, Per Ounce | | |
|---|---|---|---|---|---|---|---|---|
| *Oz.* | *Dwts.* | *Grs.* | *Carats.* | *Grs.* | *Eighths.* | *£* | *s.* | *d.* |
| 0 | 0 | 18·125 | 0 | 3 | 5 | 0 | 3 | 2·4950 |
| 0 | 0 | 17·500 | 0 | 3 | 4 | 0 | 3 | 1·1676 |
| 0 | 0 | 16·875 | 0 | 3 | 3 | 0 | 2 | 11·8401 |
| 0 | 0 | 16·250 | 0 | 3 | 2 | 0 | 2 | 10·5127 |
| 0 | 0 | 15·625 | 0 | 3 | 1 | 0 | 2 | 9·1853 |
| 0 | 0 | 15·000 | 0 | 3 | 0 | 0 | 2 | 7·8579 |
| 0 | 0 | 14·375 | 0 | 2 | 7 | 0 | 2 | 6·5305 |
| 0 | 0 | 13·750 | 0 | 2 | 6 | 0 | 2 | 5·2031 |
| 0 | 0 | 13·125 | 0 | 2 | 5 | 0 | 2 | 3·8757 |
| 0 | 0 | 12·500 | 0 | 2 | 4 | 0 | 2 | 2·5482 |
| 0 | 0 | 11·875 | 0 | 2 | 3 | 0 | 2 | 1·2208 |
| 0 | 0 | 11·250 | 0 | 2 | 2 | 0 | 1 | 11·8934 |
| 0 | 0 | 10·625 | 0 | 2 | 1 | 0 | 1 | 10·5660 |
| 0 | 0 | 10·000 | 0 | 2 | 0 | 0 | 1 | 9·2386 |
| 0 | 0 | 9·375 | 0 | 1 | 7 | 0 | 1 | 7·9112 |
| 0 | 0 | 8·750 | 0 | 1 | 6 | 0 | 1 | 6·5838 |
| 0 | 0 | 8·125 | 0 | 1 | 5 | 0 | 1 | 5·2563 |
| 0 | 0 | 7·500 | 0 | 1 | 4 | 0 | 1 | 3·9289 |
| 0 | 0 | 6·875 | 0 | 1 | 3 | 0 | 1 | 2·6015 |
| 0 | 0 | 6·250 | 0 | 1 | 2 | 0 | 1 | 1·2741 |
| 0 | 0 | 5·625 | 0 | 1 | 1 | 0 | 0 | 11·9467 |
| 0 | 0 | 5·000 | 0 | 1 | 0 | 0 | 0 | 10·6193 |
| 0 | 0 | 4·375 | 0 | 0 | 7 | 0 | 0 | 9·2919 |
| 0 | 0 | 3·750 | 0 | 0 | 6 | 0 | 0 | 7·9644 |
| 0 | 0 | 3·125 | 0 | 0 | 5 | 0 | 0 | 6·6370 |
| 0 | 0 | 2·500 | 0 | 0 | 4 | 0 | 0 | 5·3096 |
| 0 | 0 | 1·875 | 0 | 0 | 3 | 0 | 0 | 3·9822 |
| 0 | 0 | 1·250 | 0 | 0 | 2 | 0 | 0 | 2·6548 |
| 0 | 0 | 0·625 | 0 | 0 | 1 | 0 | 0 | 1·3274 |

To convert MINT VALUE into BANK VALUE when the Standard is expressed in Carats, Grains, and Eighths. This can be readily accomplished for every report by the following Tables:—

### TABLE A.

| CARATS | VALUE IN PENCE | CARATS | VALUE IN PENCE |
|---|---|---|---|
| 1 | ·0681 | 13 | ·8863 |
| 2 | ·1363 | 14 | ·9545 |
| 3 | ·2045 | 15 | 1·0227 |
| 4 | ·2727 | 16 | 1·0909 |
| 5 | ·3409 | 17 | 1·1590 |
| 6 | ·4090 | 18 | 1·2272 |
| 7 | ·4772 | 19 | 1·2954 |
| 8 | ·5454 | 20 | 1·3636 |
| 9 | ·6136 | 21 | 1·4318 |
| 10 | ·6818 | 22 | 1·5000 |
| 11 | ·7500 | 23 | 1·5681 |
| 12 | ·8181 | 24 | 1·6363 |

### TABLE B.

| CARAT GRAINS | VALUE IN PENCE | CARAT GRAINS | VALUE IN PENCE |
|---|---|---|---|
| 1 | ·0170 | 3 | ·0511 |
| 2 | ·0340 | 4 | ·0681 |

### TABLE C.

| EIGHTH CARAT GRAINS | VALUE IN PENCE | EIGHTH CARAT GRAINS | VALUE IN PENCE |
|---|---|---|---|
| 1 | ·0021 | 5 | ·0106 |
| 3 | ·0042 | 6 | ·0127 |
| 3 | ·0063 | 7 | ·0149 |
| 4 | ·0085 | 8 | ·0170 |

Table A gives the difference in price between Mint and Bank value for each carat up to fine gold; Table B the same for carat grains; and Table C the same for eighths of carat grains.

Now as the Bank value of gold is £3 17*s.* 9*d.* per oz. standard against Mint value of £3 17*s.* 10½*d.*, it follows by calculation that fine gold would fetch, Bank price, only £4 4*s.* 9·8182*d.*, instead of £4 4*s.* 11·4545*d.*, as shown by Table I. of Mint Values; and the Bank value of 1 oz. of gold, of any standard whatever, may be readily ascertained by the above Tables A, B, and C, and Table I.—the Tables A, B, and C, giving the quantities in pence to be deducted from the corresponding standard in Table I. Thus, suppose it is necessary to ascertain the Bank value of 1 oz. of gold of 14 carats 2 grains 5 eighths fine: refer to Table A, at 14 carats is found ·9545*d.*; at 2 grains in Table B is found ·0340*d.*; and at 5 eighths in Table C ·0106*d.* Now ·9545 + ·0340 + ·0106 = ·9991, which has to be deducted from £2 11*s.* 10·5575*d.* (see Table I.), leaving £2 11*s.* 9·5564*d.* as the Bank value of 1 oz. of gold of the above fineness.

## TABLE II.

TABLE of relative proportions of FINE GOLD and ALLOY, with the respective Mint Values of 1 oz. of each Alloy when the Standard is expressed in Thousandths.

| FINE GOLD | ALLOY | VALUE | | | FINE GOLD | ALLOY | VALUE | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | *s.* | *d.* | | | £ | *s.* | *d.* |
| 1000 | ·000 | 4 | 4 | 11·4545 | 986 | ·014 | 4 | 3 | 9·1821 |
| 999 | ·001 | 4 | 4 | 10·4350 | 985 | ·015 | 4 | 3 | 8·1627 |
| 998 | ·002 | 4 | 4 | 9·4156 | 984 | ·016 | 4 | 3 | 7·1432 |
| 997 | ·003 | 4 | 4 | 8·3961 | 983 | ·017 | 4 | 3 | 6·1238 |
| 996 | ·004 | 4 | 4 | 7·3767 | 982 | ·018 | 4 | 3 | 5·1043 |
| 995 | ·005 | 4 | 4 | 6·3572 | 981 | ·019 | 4 | 3 | 4·0849 |
| 994 | ·006 | 4 | 4 | 5·3378 | 980 | ·020 | 4 | 3 | 3·0654 |
| 993 | ·007 | 4 | 4 | 4·3183 | 979 | ·021 | 4 | 3 | 2·0459 |
| 992 | ·008 | 4 | 4 | 3·2989 | 978 | ·022 | 4 | 3 | 1·0265 |
| 991 | ·009 | 4 | 4 | 2·2793 | 977 | ·023 | 4 | 3 | 0·0070 |
| 990 | ·010 | 4 | 4 | 1·2600 | 976 | ·024 | 4 | 2 | 10·9876 |
| 989 | ·011 | 4 | 4 | 0·2405 | 975 | ·025 | 4 | 2 | 9·9681 |
| 988 | ·012 | 4 | 3 | 11·2210 | 974 | ·026 | 4 | 2 | 8·9487 |
| 987 | ·013 | 4 | 3 | 10·2016 | 973 | ·027 | 4 | 2 | 7·9292 |

| FINE GOLD | ALLOY | VALUE | | | FINE GOLD | ALLOY | VALUE | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 972 | ·028 | 4 | 2 | 6·9098 | 929 | ·071 | 3 | 18 | 11·0732 |
| 971 | ·029 | 4 | 2 | 5·8903 | 928 | ·072 | 3 | 18 | 10·0538 |
| 970 | ·030 | 4 | 2 | 4·8709 | 927 | ·073 | 3 | 18 | 9·0343 |
| 969 | ·031 | 4 | 2 | 3·8504 | 926 | ·074 | 3 | 18 | 8·0149 |
| 968 | ·032 | 4 | 2 | 2·8319 | 925 | ·075 | 3 | 18 | 6·9954 |
| 967 | ·033 | 4 | 2 | 1·8125 | 924 | ·076 | 3 | 18 | 5·9759 |
| 966 | ·034 | 4 | 2 | 0·7930 | 923 | ·077 | 3 | 18 | 4·9565 |
| 965 | ·035 | 4 | 1 | 11·7736 | 922 | ·078 | 3 | 18 | 3·9370 |
| 964 | ·036 | 4 | 1 | 10·7541 | 921 | ·079 | 3 | 18 | 2·9176 |
| 963 | ·037 | 4 | 1 | 9·7347 | 920 | ·080 | 3 | 18 | 1·8981 |
| 962 | ·038 | 4 | 1 | 8·7152 | 919 | ·081 | 3 | 18 | 0·8787 |
| 961 | ·039 | 4 | 1 | 7·6958 | 918 | ·082 | 3 | 17 | 11·8592 |
| 960 | ·040 | 4 | 1 | 6·6763 | 917 | ·083 | 3 | 17 | 10·8398 |
| 959 | ·041 | 4 | 1 | 5·6569 | 916* | ·084 | 3 | 17 | 9·8203 |
| 958 | ·042 | 4 | 1 | 4·6374 | 915 | ·085 | 3 | 17 | 8·8009 |
| 957 | ·043 | 4 | 1 | 3·6179 | 914 | ·086 | 3 | 17 | 7·7814 |
| 956 | ·044 | 4 | 1 | 2·5985 | 913 | ·087 | 3 | 17 | 6·7619 |
| 955 | ·045 | 4 | 1 | 1·5790 | 912 | ·088 | 3 | 17 | 5·7425 |
| 954 | ·046 | 4 | 1 | 0·5596 | 911 | ·089 | 3 | 17 | 4·7230 |
| 953 | ·047 | 4 | 0 | 11·5401 | 910 | ·090 | 3 | 17 | 3·7036 |
| 952 | ·048 | 4 | 0 | 10·5207 | 909 | ·091 | 3 | 17 | 2·6841 |
| 951 | ·049 | 4 | 0 | 9·5012 | 908 | ·092 | 3 | 17 | 1·6647 |
| 950 | ·050 | 4 | 0 | 8·4818 | 907 | ·093 | 3 | 17 | 0·6452 |
| 949 | ·051 | 4 | 0 | 7·4623 | 906 | ·094 | 3 | 16 | 11·6258 |
| 948 | ·052 | 4 | 0 | 6·4429 | 905 | ·095 | 3 | 16 | 10·6063 |
| 947 | ·053 | 4 | 0 | 5·4234 | 904 | ·096 | 3 | 16 | 9·5869 |
| 946 | ·054 | 4 | 0 | 4·4039 | 903 | ·097 | 3 | 16 | 8·5674 |
| 945 | ·055 | 4 | 0 | 3·3835 | 902 | ·098 | 3 | 16 | 7·5479 |
| 944 | ·056 | 4 | 0 | 2·3650 | 901 | ·099 | 3 | 16 | 6·5285 |
| 943 | ·057 | 4 | 0 | 1·3456 | 900 | ·100 | 3 | 16 | 5·5090 |
| 942 | ·058 | 4 | 0 | 0·3261 | 899 | ·101 | 3 | 16 | 4·4896 |
| 941 | ·059 | 3 | 19 | 11·3067 | 898 | ·102 | 3 | 16 | 3·4701 |
| 940 | ·060 | 3 | 19 | 10·2872 | 897 | ·103 | 3 | 16 | 2·4507 |
| 939 | ·061 | 3 | 19 | 9·2678 | 896 | ·104 | 3 | 16 | 1·4312 |
| 938 | ·062 | 3 | 19 | 8·2483 | 895 | ·105 | 3 | 16 | 0·4118 |
| 937 | ·063 | 3 | 19 | 7·2289 | 894 | ·106 | 3 | 15 | 11·3923 |
| 936 | ·064 | 3 | 19 | 6·2094 | 893 | ·107 | 3 | 15 | 10·3729 |
| 935 | ·065 | 3 | 19 | 5·1899 | 892 | ·108 | 3 | 15 | 9·3534 |
| 934 | ·066 | 3 | 19 | 4·1705 | 891 | ·109 | 3 | 15 | 8·3339 |
| 933 | ·067 | 3 | 19 | 3·1510 | 890 | ·110 | 3 | 15 | 7·3145 |
| 932 | ·068 | 3 | 19 | 2·1316 | 889 | ·111 | 3 | 15 | 6·2950 |
| 931 | ·069 | 3 | 19 | 1·1121 | 888 | ·112 | 3 | 15 | 5·2756 |
| 930 | ·070 | 3 | 19 | 0·0927 | 887 | ·113 | 3 | 15 | 4·2561 |

* 916·666 Standard ·083·333 £3 17*s.* 10·5000*d.*

| Fine Gold | Alloy | Value |  |  | Fine Gold | Alloy | Value |  |  |
|---|---|---|---|---|---|---|---|---|---|
|  |  | £ | *s.* | *d.* |  |  | £ | *s.* | *d.* |
| 886 | ·114 | 3 | 15 | 3·2367 | 841 | ·159 | 3 | 11 | 5·3612 |
| 885 | ·115 | 3 | 15 | 2·2172 | 840 | ·160 | 3 | 11 | 4·3418 |
| 884 | ·116 | 3 | 15 | 1·1978 | 839 | ·161 | 3 | 11 | 3·3223 |
| 883 | ·117 | 3 | 15 | 0·1783 | 838 | ·162 | 3 | 11 | 2·3029 |
| 882 | ·118 | 3 | 14 | 11·1589 | 837 | ·163 | 3 | 11 | 1·3834 |
| 881 | ·119 | 3 | 14 | 10·1394 | 836 | ·164 | 3 | 11 | 0·2639 |
| 880 | ·120 | 3 | 14 | 9·1199 | 835 | ·165 | 3 | 10 | 11·2445 |
| 879 | ·121 | 3 | 14 | 8·1005 | 834 | ·166 | 3 | 10 | 10·2250 |
| 878 | ·122 | 3 | 14 | 7·0810 | 833 | ·167 | 3 | 10 | 9·2056 |
| 877 | ·123 | 3 | 14 | 6·0616 | 832 | ·168 | 3 | 10 | 8·1861 |
| 876 | ·124 | 3 | 14 | 5·0421 | 831 | ·169 | 3 | 10 | 7·1667 |
| 875 | ·125 | 3 | 14 | 4·0227 | 830 | ·170 | 3 | 10 | 6·1472 |
| 874 | ·126 | 3 | 14 | 3·0032 | 829 | ·171 | 3 | 10 | 5·1278 |
| 873 | ·127 | 3 | 14 | 1·9838 | 828 | ·172 | 3 | 10 | 4·1083 |
| 872 | ·128 | 3 | 14 | 0·9643 | 827 | ·173 | 3 | 10 | 3·0889 |
| 871 | ·129 | 3 | 13 | 11·9449 | 826 | ·174 | 3 | 10 | 2·0694 |
| 870 | ·130 | 3 | 13 | 10·9254 | 825 | ·175 | 3 | 10 | 1·0499 |
| 869 | ·131 | 3 | 13 | 9·9059 | 824 | ·176 | 3 | 10 | 0·0305 |
| 868 | ·132 | 3 | 13 | 8·8865 | 823 | ·177 | 3 | 9 | 11·0110 |
| 867 | ·133 | 3 | 13 | 7·8670 | 822 | ·178 | 3 | 9 | 9·9916 |
| 866 | ·134 | 3 | 13 | 6·8476 | 821 | ·179 | 3 | 9 | 8·9721 |
| 865 | ·135 | 3 | 13 | 5·8281 | 820 | ·180 | 3 | 9 | 7·9527 |
| 864 | ·136 | 3 | 13 | 4·8087 | 819 | ·181 | 3 | 9 | 6·9332 |
| 863 | ·137 | 3 | 13 | 3·7892 | 818 | ·182 | 3 | 9 | 5·9138 |
| 862 | ·138 | 3 | 13 | 2·7698 | 817 | ·183 | 3 | 9 | 4·8943 |
| 861 | ·139 | 3 | 13 | 1·7503 | 816 | ·184 | 3 | 9 | 3·8749 |
| 860 | ·140 | 3 | 13 | 0·7309 | 815 | ·185 | 3 | 9 | 2·8554 |
| 859 | ·141 | 3 | 12 | 11·7114 | 814 | ·186 | 3 | 9 | 1·8359 |
| 858 | ·142 | 3 | 12 | 10·6919 | 813 | ·187 | 3 | 9 | 0·8165 |
| 857 | ·143 | 3 | 12 | 9·6725 | 812 | ·188 | 3 | 8 | 11·7970 |
| 856 | ·144 | 3 | 12 | 8·6530 | 811 | ·189 | 3 | 8 | 10·7776 |
| 855 | ·145 | 3 | 12 | 7·6336 | 810 | ·190 | 3 | 8 | 9·7581 |
| 854 | ·146 | 3 | 12 | 6·6141 | 809 | ·191 | 3 | 8 | 8·7387 |
| 853 | ·147 | 3 | 12 | 5·5947 | 808 | ·192 | 3 | 8 | 7·7192 |
| 852 | ·148 | 3 | 12 | 4·5752 | 807 | ·193 | 3 | 8 | 6·6998 |
| 851 | ·149 | 3 | 12 | 3·5558 | 806 | ·194 | 3 | 8 | 5·6803 |
| 850 | ·150 | 3 | 12 | 2·5363 | 805 | ·195 | 3 | 8 | 4·6609 |
| 849 | ·151 | 3 | 12 | 1·5169 | 804 | ·196 | 3 | 8 | 3·6414 |
| 848 | .152 | 3 | 12 | 0·4974 | 803 | ·197 | 3 | 8 | 2·6219 |
| 847 | ·153 | 3 | 11 | 11·4779 | 802 | ·198 | 3 | 8 | 1·6025 |
| 846 | ·154 | 3 | 11 | 10·4585 | 801 | ·199 | 3 | 8 | 0·5830 |
| 845 | ·155 | 3 | 11 | 9·4390 | 800 | ·200 | 3 | 7 | 11·5636 |
| 844 | ·156 | 3 | 11 | 8·4196 | 799 | ·201 | 3 | 7 | 10·5441 |
| 843 | ·157 | 3 | 11 | 7·4001 | 798 | ·202 | 3 | 7 | 9·5247 |
| 842 | ·158 | 3 | 11 | 6·3807 | 797 | ·203 | 3 | 7 | 8·5052 |

| Fine Gold | Alloy | Value | | | Fine Gold | Alloy | Value | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 796 | ·204 | 3 | 7 | 7·4858 | 751 | ·249 | 3 | 3 | 9·6103 |
| 795 | ·205 | 3 | 7 | 6·4663 | 750 | ·250 | 3 | 3 | 8·5909 |
| 794 | ·206 | 3 | 7 | 5·4469 | 749 | ·251 | 3 | 3 | 7·5714 |
| 793 | ·207 | 3 | 7 | 4·4274 | 748 | ·252 | 3 | 3 | 6·5519 |
| 792 | ·208 | 3 | 7 | 3·4979 | 747 | ·253 | 3 | 3 | 5·5325 |
| 791 | ·209 | 3 | 7 | 2·3885 | 746 | ·254 | 3 | 3 | 4·5130 |
| 790 | ·210 | 3 | 7 | 1·3690 | 745 | ·255 | 3 | 3 | 3·4936 |
| 789 | ·211 | 3 | 7 | 0·3496 | 744 | ·256 | 3 | 3 | 2·4741 |
| 788 | ·212 | 3 | 6 | 11·3301 | 743 | ·257 | 3 | 3 | 1·4547 |
| 787 | ·213 | 3 | 6 | 10·3107 | 742 | ·258 | 3 | 3 | 0·4352 |
| 786 | ·214 | 3 | 6 | 9·2912 | 741 | ·259 | 3 | 2 | 11·4158 |
| 785 | ·215 | 3 | 6 | 8·2718 | 740 | ·260 | 3 | 2 | 10·3963 |
| 784 | ·216 | 3 | 6 | 7·2523 | 739 | ·261 | 3 | 2 | 9·3769 |
| 783 | ·217 | 3 | 6 | 6·2329 | 738 | ·262 | 3 | 2 | 8·3574 |
| 782 | ·218 | 3 | 6 | 5·2134 | 737 | ·263 | 3 | 2 | 7·3379 |
| 781 | ·219 | 3 | 6 | 4·1939 | 736 | ·264 | 3 | 2 | 6·3185 |
| 780 | ·220 | 3 | 6 | 3·1745 | 735 | ·265 | 3 | 2 | 5·2990 |
| 779 | ·221 | 3 | 6 | 2·1550 | 734 | ·266 | 3 | 2 | 4·2796 |
| 778 | ·222 | 3 | 6 | 1·1356 | 733 | ·267 | 3 | 2 | 3·2601 |
| 777 | ·223 | 3 | 6 | 0·1161 | 732 | ·268 | 3 | 2 | 2·2407 |
| 776 | ·224 | 3 | 5 | 11·0967 | 731 | ·269 | 3 | 2 | 1·2212 |
| 775 | ·225 | 3 | 5 | 10·0772 | 730 | ·270 | 3 | 2 | 0·2018 |
| 774 | ·226 | 8 | 5 | 9·0578 | 729 | ·271 | 3 | 1 | 11·1823 |
| 773 | ·227 | 3 | 5 | 8·0383 | 728 | ·272 | 3 | 1 | 10·1629 |
| 772 | ·228 | 3 | 5 | 7·0189 | 727 | ·273 | 3 | 1 | 9·1434 |
| 771 | ·229 | 3 | 5 | 5·9994 | 726 | ·274 | 3 | 1 | 8·1239 |
| 770 | ·230 | 3 | 5 | 4·9799 | 725 | ·275 | 3 | 1 | 7·1045 |
| 769 | ·231 | 3 | 5 | 3·9605 | 724 | ·276 | 3 | 1 | 6·0850 |
| 768 | ·232 | 3 | 5 | 2·9410 | 723 | ·277 | 3 | 1 | 5·0656 |
| 767 | ·233 | 3 | 5 | 1·9216 | 722 | ·278 | 3 | 1 | 4·0461 |
| 766 | ·234 | 3 | 5 | 0·9021 | 721 | ·279 | 3 | 1 | 3·0267 |
| 765 | ·235 | 3 | 4 | 11·8827 | 720 | ·280 | 3 | 1 | 2·0072 |
| 764 | ·236 | 3 | 4 | 10·8632 | 719 | ·281 | 3 | 1 | 0·9878 |
| 763 | ·237 | 3 | 4 | 9·8438 | 718 | ·282 | 3 | 0 | 11·9683 |
| 762 | ·238 | 3 | 4 | 8·8243 | 717 | ·283 | 3 | 0 | 10·9489 |
| 761 | ·239 | 3 | 4 | 7·8049 | 716 | ·284 | 3 | 0 | 9·9294 |
| 760 | ·240 | 3 | 4 | 6·7854 | 715 | ·285 | 3 | 0 | 8·9099 |
| 759 | ·241 | 3 | 4 | 5·7659 | 714 | ·286 | 3 | 0 | 7·8905 |
| 758 | ·242 | 3 | 4 | 4·7465 | 713 | ·287 | 3 | 0 | 6·8710 |
| 757 | ·243 | 3 | 4 | 3·7270 | 712 | ·288 | 3 | 0 | 5·8516 |
| 756 | ·244 | 3 | 4 | 2·7076 | 711 | ·289 | 3 | 0 | 4·8321 |
| 755 | ·245 | 3 | 4 | 1·6881 | 710 | ·290 | 3 | 0 | 3·8127 |
| 754 | ·246 | 3 | 4 | 0·6687 | 709 | ·291 | 3 | 0 | 2·7932 |
| 753 | ·247 | 3 | 3 | 11·6492 | 708 | ·292 | 3 | 0 | 1·7738 |
| 752 | ·248 | 3 | 3 | 10·6298 | 707 | ·293 | 3 | 0 | 0·7543 |

| Fine Gold | Alloy | Value | | | Fine Gold | Alloy | Value | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 706 | ·294 | 2 | 19 | 11·7349 | 661 | ·339 | 2 | 16 | 1·8594 |
| 705 | ·295 | 2 | 19 | 10·7154 | 660 | ·340 | 2 | 16 | 0·8399 |
| 704 | ·296 | 2 | 19 | 9·6959 | 659 | ·341 | 2 | 15 | 11·8205 |
| 703 | ·297 | 2 | 19 | 8·6765 | 658 | ·342 | 2 | 15 | 10·8010 |
| 702 | ·298 | 2 | 19 | 7·6570 | 657 | ·343 | 2 | 15 | 9·7816 |
| 701 | ·299 | 2 | 19 | 6·6376 | 656 | ·344 | 2 | 15 | 8·7621 |
| 700 | ·300 | 2 | 19 | 5·6181 | 655 | ·345 | 2 | 15 | 7·7427 |
| 699 | ·301 | 2 | 19 | 4·5987 | 654 | ·346 | 2 | 15 | 6·7232 |
| 698 | ·302 | 2 | 19 | 3·5792 | 653 | ·347 | 2 | 15 | 5·7038 |
| 697 | ·303 | 2 | 19 | 2·5598 | 652 | ·348 | 2 | 15 | 4·6843 |
| 696 | ·304 | 2 | 19 | 1·5403 | 651 | ·349 | 2 | 15 | 3·6649 |
| 695 | ·305 | 2 | 19 | 0·5209 | 650 | ·350 | 2 | 15 | 2·6454 |
| 694 | ·306 | 2 | 18 | 11·5014 | 649 | ·351 | 2 | 15 | 1·6259 |
| 693 | ·307 | 2 | 18 | 10·4820 | 648 | ·352 | 2 | 15 | 0·6065 |
| 692 | ·308 | 2 | 18 | 9·4625 | 647 | ·353 | 2 | 14 | 11·5870 |
| 691 | ·309 | 2 | 18 | 8·4430 | 646 | ·354 | 2 | 14 | 10·5676 |
| 690 | ·310 | 2 | 18 | 7·4236 | 645 | ·355 | 2 | 14 | 9·5481 |
| 689 | ·311 | 2 | 18 | 6·4041 | 644 | ·356 | 2 | 14 | 8·5287 |
| 688 | ·312 | 2 | 18 | 5·3847 | 643 | ·357 | 2 | 14 | 7·5092 |
| 687 | ·313 | 2 | 18 | 4·3652 | 642 | ·358 | 2 | 14 | 6·4898 |
| 686 | ·314 | 2 | 18 | 3·3458 | 641 | ·359 | 2 | 14 | 5·4703 |
| 685 | ·315 | 2 | 18 | 2·3263 | 640 | ·360 | 2 | 14 | 4·4509 |
| 684 | ·316 | 2 | 18 | 1·3069 | 639 | ·361 | 2 | 14 | 3·4314 |
| 683 | ·317 | 3 | 18 | 0·2874 | 638 | ·362 | 2 | 14 | 2·4120 |
| 682 | ·318 | 2 | 17 | 11·2680 | 637 | ·363 | 2 | 14 | 1·3925 |
| 681 | ·319 | 2 | 17 | 10·2485 | 636 | ·364 | 2 | 14 | 0·3730 |
| 680 | ·320 | 2 | 17 | 9·2290 | 635 | ·365 | 2 | 13 | 11·3536 |
| 679 | ·321 | 2 | 17 | 8·2096 | 634 | ·366 | 2 | 13 | 10·3341 |
| 678 | ·322 | 2 | 17 | 7·1901 | 633 | ·367 | 2 | 13 | 9·3147 |
| 677 | ·323 | 2 | 17 | 6·1707 | 632 | ·368 | 2 | 13 | 8·2952 |
| 676 | ·324 | 2 | 17 | 5·1512 | 631 | ·369 | 2 | 13 | 7·2758 |
| 675 | ·325 | 2 | 17 | 4·1318 | 630 | ·370 | 2 | 13 | 6·2563 |
| 674 | ·326 | 2 | 17 | 3·1123 | 629 | ·371 | 2 | 13 | 5·2369 |
| 673 | ·327 | 2 | 17 | 2·0929 | 628 | ·372 | 2 | 13 | 4·2174 |
| 672 | ·328 | 2 | 17 | 1·0734 | 627 | ·373 | 2 | 13 | 3·1979 |
| 671 | ·329 | 2 | 17 | 0·0540 | 626 | ·374 | 2 | 13 | 2·1785 |
| 670 | ·330 | 2 | 16 | 11·0345 | 625 | ·375 | 2 | 13 | 1·1590 |
| 669 | ·331 | 2 | 16 | 10·0151 | 624 | ·376 | 2 | 13 | 0·1396 |
| 668 | ·332 | 2 | 16 | 8·9956 | 623 | ·377 | 2 | 12 | 11·1201 |
| 667 | ·333 | 2 | 16 | 7·9761 | 622 | ·378 | 2 | 12 | 10·1007 |
| 666 | ·334 | 2 | 16 | 6·9567 | 621 | ·379 | 2 | 12 | 9·0812 |
| 665 | ·335 | 2 | 16 | 5·9372 | 620 | ·380 | 2 | 12 | 8·0618 |
| 664 | ·336 | 2 | 16 | 4·9178 | 619 | ·381 | 2 | 12 | 7·0423 |
| 663 | ·337 | 2 | 16 | 3·8983 | 618 | ·382 | 2 | 12 | 6·0229 |
| 662 | ·338 | 2 | 16 | 2·8789 | 617 | ·383 | 2 | 12 | 5·0034 |

| Fine Gold | Alloy | Value | | | Fine Gold | Alloy | Value | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 616 | ·384 | 2 | 12 | 3·9839 | 571 | ·429 | 2 | 8 | 6·1085 |
| 615 | ·385 | 2 | 12 | 2·9645 | 570 | ·430 | 2 | 8 | 5·0890 |
| 614 | ·386 | 2 | 12 | 1·9451 | 569 | ·431 | 2 | 8 | 4·0696 |
| 613 | ·387 | 2 | 12 | 0·9256 | 568 | ·432 | 2 | 8 | 3·0501 |
| 612 | ·388 | 2 | 11 | 11·9061 | 567 | ·433 | 2 | 8 | 2·0307 |
| 611 | ·389 | 2 | 11 | 10·8867 | 566 | ·434 | 2 | 8 | 1·0112 |
| 610 | ·390 | 2 | 11 | 9·8672 | 565 | ·435 | 2 | 7 | 11·9918 |
| 609 | ·391 | 2 | 11 | 8·8478 | 564 | ·436 | 2 | 7 | 10·9723 |
| 608 | ·392 | 2 | 11 | 7·8283 | 563 | ·437 | 2 | 7 | 9·9529 |
| 607 | ·393 | 2 | 11 | 6·8089 | 562 | ·438 | 2 | 7 | 8·9334 |
| 606 | ·394 | 2 | 11 | 5·7894 | 561 | ·439 | 2 | 7 | 7·9140 |
| 605 | ·395 | 2 | 11 | 4·7699 | 560 | ·440 | 2 | 7 | 6·8945 |
| 604 | ·396 | 2 | 11 | 3·7505 | 559 | ·441 | 2 | 7 | 5·8751 |
| 603 | ·397 | 2 | 11 | 2·7311 | 558 | ·442 | 2 | 7 | 4·8556 |
| 602 | ·398 | 2 | 11 | 1·7116 | 557 | ·443 | 2 | 7 | 3·8361 |
| 601 | ·399 | 2 | 11 | 0·6921 | 556 | ·444 | 2 | 7 | 2·8167 |
| 600 | ·400 | 2 | 10 | 11·6727 | 555 | ·445 | 2 | 7 | 1·7972 |
| 599 | ·401 | 2 | 10 | 10·6532 | 554 | ·446 | 2 | 7 | 0·7778 |
| 598 | ·402 | 2 | 10 | 9·6338 | 553 | ·447 | 2 | 6 | 11·7583 |
| 597 | ·403 | 2 | 10 | 8·6143 | 552 | ·448 | 2 | 6 | 10·7389 |
| 596 | ·404 | 2 | 10 | 7·5949 | 551 | ·449 | 2 | 6 | 9·7194 |
| 595 | ·405 | 2 | 10 | 6·5754 | 550 | ·450 | 2 | 6 | 8·6999 |
| 594 | ·406 | 2 | 10 | 5·5559 | 549 | ·451 | 2 | 6 | 7·6805 |
| 593 | ·407 | 2 | 10 | 4·5365 | 548 | ·452 | 2 | 6 | 6·6611 |
| 592 | ·408 | 2 | 10 | 3·5170 | 547 | ·453 | 2 | 6 | 5·6416 |
| 591 | ·409 | 2 | 10 | 2·4976 | 546 | ·454 | 2 | 6 | 4·6221 |
| 590 | ·410 | 2 | 10 | 1·4781 | 545 | ·455 | 2 | 6 | 3·6027 |
| 589 | ·411 | 2 | 10 | 0·4587 | 544 | ·456 | 2 | 6 | 2·5832 |
| 588 | ·412 | 2 | 9 | 11·4392 | 543 | ·457 | 2 | 6 | 1·5638 |
| 587 | ·413 | 2 | 9 | 10·4198 | 542 | ·458 | 2 | 6 | 0·5443 |
| 586 | ·414 | 2 | 9 | 9·4003 | 541 | ·459 | 2 | 5 | 11·5249 |
| 685 | ·415 | 2 | 9 | 8·3809 | 540 | ·460 | 2 | 5 | 10·5054 |
| 584 | ·416 | 2 | 9 | 7·3614 | 539 | ·461 | 2 | 5 | 9·4859 |
| 583 | ·417 | 2 | 9 | 6·3419 | 538 | ·462 | 2 | 5 | 8·4665 |
| 582 | ·418 | 2 | 9 | 5·3225 | 537 | ·463 | 2 | 5 | 7·4470 |
| 581 | ·419 | 2 | 9 | 4·3030 | 536 | ·464 | 2 | 5 | 6·4276 |
| 580 | ·420 | 2 | 9 | 3·2836 | 535 | ·465 | 2 | 5 | 5·4081 |
| 579 | ·421 | 2 | 9 | 2·2641 | 534 | ·466 | 2 | 5 | 4·3887 |
| 578 | ·422 | 2 | 9 | 1·2447 | 533 | ·467 | 2 | 5 | 3·3692 |
| 577 | ·423 | 2 | 9 | 0·2252 | 532 | ·468 | 2 | 5 | 2·3498 |
| 576 | ·424 | 2 | 8 | 11·2058 | 531 | ·469 | 2 | 5 | 1·3303 |
| 575 | ·425 | 2 | 8 | 10·1863 | 530 | ·470 | 2 | 5 | 0·3109 |
| 574 | ·426 | 2 | 8 | 9·1669 | 529 | ·471 | 2 | 4 | 11·2914 |
| 573 | ·427 | 2 | 8 | 8·1474 | 528 | ·472 | 2 | 4 | 10·2719 |
| 572 | ·428 | 2 | 8 | 7·1279 | 527 | ·473 | 2 | 4 | 9·2525 |

| Fine Gold | Alloy | Value | | | Fine Gold | Alloy | Value | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 526 | ·474 | 2 | 4 | 8·2330 | 481 | ·519 | 2 | 0 | 10·3576 |
| 525 | ·475 | 2 | 4 | 7·2136 | 480 | ·520 | 2 | 0 | 9·3381 |
| 524 | ·476 | 2 | 4 | 6·1941 | 479 | ·521 | 2 | 0 | 8·3187 |
| 523 | ·477 | 2 | 4 | 5·1747 | 478 | ·522 | 2 | 0 | 7·2992 |
| 522 | ·478 | 2 | 4 | 4·1552 | 477 | ·523 | 2 | 0 | 6·2798 |
| 521 | ·479 | 2 | 4 | 3·1358 | 476 | ·524 | 2 | 0 | 5·2603 |
| 520 | ·480 | 2 | 4 | 2·1163 | 475 | ·525 | 2 | 0 | 4·2409 |
| 519 | ·481 | 2 | 4 | 1·0969 | 474 | ·526 | 2 | 0 | 3·2214 |
| 518 | ·482 | 2 | 4 | 0·0774 | 473 | ·527 | 2 | 0 | 2·2020 |
| 517 | ·483 | 2 | 3 | 11·0579 | 472 | ·528 | 2 | 0 | 1·1825 |
| 516 | ·484 | 2 | 3 | 10·0385 | 471 | ·529 | 2 | 0 | 0·1630 |
| 515 | ·485 | 2 | 3 | 9·0190 | 470 | ·530 | 1 | 19 | 11·1436 |
| 514 | ·486 | 2 | 3 | 7·9996 | 469 | ·531 | 1 | 19 | 10·1241 |
| 513 | ·487 | 2 | 3 | 6·9801 | 468 | ·532 | 1 | 19 | 9·1047 |
| 512 | ·488 | 2 | 3 | 5·9607 | 467 | ·533 | 1 | 19 | 8·0852 |
| 511 | ·489 | 2 | 3 | 4·9412 | 466 | ·534 | 1 | 19 | 7·0658 |
| 510 | ·490 | 2 | 3 | 3·9218 | 465 | ·535 | 1 | 19 | 6·0463 |
| 509 | ·491 | 2 | 3 | 2·9023 | 464 | ·536 | 1 | 19 | 5·0269 |
| 508 | ·492 | 2 | 3 | 1·8829 | 463 | ·537 | 1 | 19 | 4·0074 |
| 507 | ·493 | 2 | 3 | 0·8634 | 462 | ·538 | 1 | 19 | 2·9879 |
| 506 | ·494 | 2 | 3 | 11·8439 | 461 | ·539 | 1 | 19 | 1·9685 |
| 505 | ·495 | 2 | 2 | 10·8245 | 460 | ·540 | 1 | 19 | 0·9490 |
| 504 | ·496 | 2 | 2 | 9·8051 | 459 | ·541 | 1 | 18 | 11·9296 |
| 503 | ·497 | 2 | 2 | 8·7856 | 458 | ·542 | 1 | 18 | 10·9101 |
| 502 | ·498 | 2 | 2 | 7·7661 | 457 | ·543 | 1 | 18 | 9·8907 |
| 501 | ·499 | 2 | 2 | 6·7467 | 456 | ·544 | 1 | 18 | 8·8712 |
| 500 | ·500 | 2 | 2 | 5·7272 | 455 | ·545 | 1 | 18 | 7·8518 |
| 499 | ·501 | 2 | 2 | 4·7078 | 454 | ·546 | 1 | 18 | 6·8323 |
| 498 | ·502 | 2 | 2 | 3·6883 | 453 | ·547 | 1 | 18 | 5·8129 |
| 497 | ·503 | 2 | 2 | 2·6689 | 452 | ·548 | 1 | 18 | 4·7934 |
| 496 | ·504 | 2 | 2 | 1·6494 | 451 | ·549 | 1 | 18 | 3·7739 |
| 495 | ·505 | 2 | 2 | 0·6300 | 450 | ·550 | 1 | 18 | 2·7545 |
| 494 | ·506 | 2 | 1 | 11·6105 | 449 | ·551 | 1 | 18 | 1·7351 |
| 493 | ·507 | 2 | 1 | 10·5911 | 448 | ·552 | 1 | 18 | 0·7156 |
| 492 | ·508 | 2 | 1 | 9·5716 | 447 | ·553 | 1 | 17 | 11·6961 |
| 491 | ·509 | 2 | 1 | 8·5521 | 446 | ·554 | 1 | 17 | 10·6767 |
| 490 | ·510 | 2 | 1 | 7·5327 | 445 | ·555 | 1 | 17 | 9·6572 |
| 489 | ·511 | 2 | 1 | 1·5132 | 444 | ·556 | 1 | 17 | 8·6378 |
| 488 | ·512 | 2 | 1 | 5·4938 | 443 | ·557 | 1 | 17 | 7·6183 |
| 487 | ·513 | 2 | 1 | 4·4743 | 442 | ·558 | 1 | 17 | 6·5989 |
| 486 | ·514 | 2 | 1 | 3·4549 | 441 | ·559 | 1 | 17 | 5·5794 |
| 485 | ·515 | 2 | 1 | 2·4354 | 440 | ·560 | 1 | 17 | 4·5599 |
| 484 | ·516 | 2 | 1 | 1·4159 | 439 | ·561 | 1 | 17 | 3·5405 |
| 483 | ·517 | 2 | 1 | 0·3965 | 438 | ·562 | 1 | 17 | 2·5211 |
| 482 | ·518 | 2 | 0 | 11·3770 | 437 | ·563 | 1 | 17 | 1·5016 |

| Fine Gold | Alloy | Value £ | s. | d. | Fine Gold | Alloy | Value £ | s. | d. |
|---|---|---|---|---|---|---|---|---|---|
| 436 | ·564 | 1 | 17 | 0·4821 | 391 | ·609 | 1 | 13 | 2·6067 |
| 435 | ·565 | 1 | 16 | 11·4627 | 390 | ·610 | 1 | 13 | 1·5872 |
| 434 | ·566 | 1 | 16 | 10·4432 | 389 | ·611 | 1 | 13 | 0·5678 |
| 433 | ·567 | 1 | 16 | 9·4238 | 388 | ·612 | 1 | 12 | 11·5483 |
| 432 | ·568 | 1 | 16 | 8·4043 | 387 | ·613 | 1 | 12 | 10·5289 |
| 431 | ·569 | 1 | 16 | 7·3849 | 386 | ·614 | 1 | 12 | 9·5094 |
| 430 | ·570 | 1 | 16 | 6·3654 | 385 | ·615 | 1 | 12 | 8·4899 |
| 429 | ·571 | 1 | 16 | 5·3459 | 384 | ·616 | 1 | 12 | 7·4705 |
| 428 | ·572 | 1 | 16 | 4·3265 | 383 | ·617 | 1 | 12 | 6·4511 |
| 427 | ·573 | 1 | 16 | 3·3070 | 382 | ·618 | 1 | 12 | 5·4316 |
| 426 | ·574 | 1 | 16 | 2·2876 | 381 | ·619 | 1 | 12 | 4·4121 |
| 425 | ·575 | 1 | 16 | 1·2681 | 380 | ·620 | 1 | 12 | 3·3927 |
| 424 | ·576 | 1 | 16 | 0·2487 | 379 | ·621 | 1 | 12 | 2·3732 |
| 423 | ·577 | 1 | 15 | 11·2292 | 378 | ·622 | 1 | 12 | 1·3538 |
| 422 | ·578 | 1 | 15 | 10·2098 | 377 | ·623 | 1 | 12 | 0·3343 |
| 421 | ·579 | 1 | 15 | 9·1903 | 376 | ·624 | 1 | 11 | 11·3142 |
| 420 | ·580 | 1 | 15 | 8·1709 | 375 | ·625 | 1 | 11 | 10·2954 |
| 419 | ·581 | 1 | 15 | 7·1514 | 374 | ·626 | 1 | 11 | 9·2759 |
| 418 | ·582 | 1 | 15 | 6·1319 | 373 | ·627 | 1 | 11 | 8·2565 |
| 417 | ·583 | 1 | 15 | 5·1125 | 372 | ·628 | 1 | 11 | 7·2370 |
| 416 | ·584 | 1 | 15 | 4·0930 | 371 | ·629 | 1 | 11 | 6·2176 |
| 415 | ·585 | 1 | 15 | 3·0736 | 370 | ·630 | 1 | 11 | 5·1981 |
| 414 | ·586 | 1 | 15 | 2·0541 | 369 | ·631 | 1 | 11 | 4·1787 |
| 413 | ·587 | 1 | 15 | 1·0347 | 368 | ·632 | 1 | 11 | 3·1592 |
| 412 | ·588 | 1 | 15 | 0·0152 | 367 | ·633 | 1 | 11 | 2·1398 |
| 411 | ·589 | 1 | 14 | 10·9958 | 366 | ·634 | 1 | 11 | 1·1203 |
| 410 | ·590 | 1 | 14 | 9·9763 | 365 | ·635 | 1 | 11 | 0·1009 |
| 409 | ·591 | 1 | 14 | 8·9569 | 364 | ·636 | 1 | 10 | 11·0814 |
| 408 | ·592 | 1 | 14 | 7·9374 | 363 | ·637 | 1 | 10 | 10·0620 |
| 407 | ·593 | 1 | 14 | 6·9179 | 362 | ·638 | 1 | 10 | 9·0425 |
| 406 | ·594 | 1 | 14 | 5·8985 | 361 | ·639 | 1 | 10 | 8·0230 |
| 405 | ·595 | 1 | 14 | 4·8790 | 360 | ·640 | 1 | 10 | 7·0036 |
| 404 | ·596 | 1 | 14 | 3·8596 | 359 | ·641 | 1 | 10 | 5·9841 |
| 403 | ·597 | 1 | 14 | 2·8401 | 358 | ·642 | 1 | 10 | 4·9647 |
| 402 | ·598 | 1 | 14 | 1·8207 | 357 | ·643 | 1 | 10 | 3·9452 |
| 401 | ·599 | 1 | 14 | 0·8012 | 356 | ·644 | 1 | 10 | 2·9258 |
| 400 | ·600 | 1 | 13 | 11·7818 | 355 | ·645 | 1 | 10 | 1·9063 |
| 399 | ·601 | 1 | 13 | 10·7623 | 354 | ·646 | 1 | 10 | 0·8869 |
| 398 | ·602 | 1 | 13 | 9·7429 | 353 | ·647 | 1 | 9 | 11·8674 |
| 397 | ·603 | 1 | 13 | 8·7234 | 352 | ·648 | 1 | 9 | 10·8479 |
| 396 | ·604 | 1 | 13 | 7·7039 | 351 | ·649 | 1 | 9 | 9·8285 |
| 395 | ·605 | 1 | 13 | 6·6845 | 350 | ·650 | 1 | 9 | 8·8090 |
| 394 | ·606 | 1 | 13 | 5·6651 | 349 | ·651 | 1 | 9 | 7·7896 |
| 393 | ·607 | 1 | 13 | 4·6456 | 348 | ·652 | 1 | 9 | 6·7701 |
| 392 | ·608 | 1 | 13 | 3·6261 | 347 | ·653 | 1 | 9 | 5·7507 |

| Fine Gold | Alloy | Value | | | Fine Gold | Alloy | Value | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 346 | ·654 | 1 | 9 | 4·7312 | 301 | ·699 | 1 | 5 | 6·8558 |
| 345 | ·655 | 1 | 9 | 3·7118 | 300 | ·700 | 1 | 5 | 5·8363 |
| 344 | ·656 | 1 | 9 | 2·6923 | 299 | ·701 | 1 | 5 | 4·8169 |
| 343 | ·657 | 1 | 9 | 1·6729 | 298 | ·702 | 1 | 5 | 3·7974 |
| 342 | ·658 | 1 | 9 | 0·6534 | 297 | ·703 | 1 | 5 | 2·7779 |
| 341 | ·659 | 1 | 8 | 11·6339 | 296 | ·704 | 1 | 5 | 1·7585 |
| 340 | ·660 | 1 | 8 | 10·6145 | 295 | ·705 | 1 | 5 | 0·7390 |
| 339 | ·661 | 1 | 8 | 9·5951 | 294 | ·706 | 1 | 4 | 11·7196 |
| 338 | ·662 | 1 | 8 | 8·5756 | 293 | ·707 | 1 | 4 | 10·7011 |
| 337 | ·663 | 1 | 8 | 7·5561 | 292 | ·708 | 1 | 4 | 9·6807 |
| 336 | ·664 | 1 | 8 | 6·5367 | 291 | ·709 | 1 | 4 | 8·6612 |
| 335 | ·665 | 1 | 8 | 5·5172 | 290 | ·710 | 1 | 4 | 7·6418 |
| 334 | ·666 | 1 | 8 | 4·4978 | 289 | ·711 | 1 | 4 | 6·6223 |
| 333 | ·667 | 1 | 8 | 3·4783 | 288 | ·712 | 1 | 4 | 5·6029 |
| 332 | ·668 | 1 | 8 | 2·4589 | 287 | ·713 | 1 | 4 | 4·5834 |
| 331 | ·669 | 1 | 8 | 1·4394 | 286 | ·714 | 1 | 4 | 3·5639 |
| 330 | ·670 | 1 | 8 | 0·4199 | 285 | ·715 | 1 | 4 | 2·5445 |
| 329 | ·671 | 1 | 7 | 11·4005 | 284 | ·716 | 1 | 4 | 1·5251 |
| 328 | ·672 | 1 | 7 | 10·3811 | 283 | ·717 | 1 | 4 | 0·5056 |
| 327 | ·673 | 1 | 7 | 9·3616 | 282 | ·718 | 1 | 3 | 11·4861 |
| 326 | ·674 | 1 | 7 | 8·3421 | 281 | ·719 | 1 | 3 | 10·4667 |
| 325 | ·675 | 1 | 7 | 7·3227 | 280 | ·720 | 1 | 3 | 9·4472 |
| 324 | ·676 | 1 | 7 | 6:3032 | 279 | ·721 | 1 | 3 | 8·4278 |
| 323 | ·677 | 1 | 7 | 5·2838 | 278 | ·722 | 1 | 3 | 7·4083 |
| 322 | ·678 | 1 | 7 | 4·2643 | 277 | ·723 | 1 | 3 | 6·3889 |
| 321 | ·679 | 1 | 7 | 3·2449 | 276 | ·724 | 1 | 3 | 5·3694 |
| 320 | ·680 | 1 | 7 | 2·2254 | 275 | ·725 | 1 | 3 | 4·3499 |
| 319 | ·681 | 1 | 7 | 1·2059 | 274 | ·726 | 1 | 3 | 3·3305 |
| 318 | ·682 | 1 | 7 | 0·1865 | 273 | ·727 | 1 | 3 | 2·3110 |
| 317 | ·683 | 1 | 6 | 11·1670 | 272 | ·728 | 1 | 3 | 1·2916 |
| 316 | ·684 | 1 | 6 | 10·1476 | 271 | ·729 | 1 | 3 | 0·2721 |
| 315 | ·685 | 1 | 6 | 9·1281 | 270 | ·730 | 1 | 2 | 11·2527 |
| 314 | ·686 | 1 | 6 | 8·1087 | 269 | ·731 | 1 | 2 | 10·2332 |
| 313 | ·687 | 1 | 6 | 7·0892 | 268 | ·732 | 1 | 2 | 9·2138 |
| 312 | ·688 | 1 | 6 | 6·0698 | 267 | ·733 | 1 | 2 | 8·1943 |
| 311 | ·689 | 1 | 6 | 5·0503 | 266 | ·234 | 1 | 2 | 7·1749 |
| 310 | ·690 | 1 | 6 | 4·0309 | 265 | ·735 | 1 | 2 | 6·1554 |
| 309 | ·691 | 1 | 6 | 3·0114 | 264 | ·736 | 1 | 2 | 5·1351 |
| 308 | ·692 | 1 | 6 | 1·9919 | 263 | ·737 | 1 | 2 | 4·1165 |
| 307 | ·693 | 1 | 6 | 0·9725 | 262 | ·738 | 1 | 2 | 3·0970 |
| 306 | ·694 | 1 | 5 | 11·9530 | 261 | ·739 | 1 | 2 | 2·0776 |
| 305 | ·695 | 1 | 5 | 10·9336 | 260 | ·740 | 1 | 2 | 1·0581 |
| 304 | ·696 | 1 | 5 | 9·9141 | 259 | ·741 | 1 | 2 | 0·0387 |
| 303 | ·697 | 1 | 5 | 8·8947 | 258 | ·742 | 1 | 1 | 11·0192 |
| 302 | ·698 | 1 | 5 | 7·8752 | 257 | ·743 | 1 | 1 | 9·9998 |

| Fine Gold | Alloy | Value | | | Fine Gold | Alloy | Value | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 256 | ·744 | 1 | 1 | 8·9803 | 211 | ·789 | 0 | 17 | 11·1049 |
| 255 | ·745 | 1 | 1 | 7·9609 | 210 | ·790 | 0 | 17 | 10·0854 |
| 254 | ·746 | 1 | 1 | 6·9414 | 209 | ·791 | 0 | 17 | 9·0659 |
| 253 | ·747 | 1 | 1 | 5·9219 | 208 | ·792 | 0 | 17 | 8·0465 |
| 252 | ·748 | 1 | 1 | 4·9025 | 207 | ·793 | 0 | 17 | 7·0270 |
| 251 | ·749 | 1 | 1 | 3·8830 | 206 | ·794 | 0 | 17 | 6·0076 |
| 250 | ·650 | 1 | 1 | 2·8636 | 205 | ·795 | 0 | 17 | 4·9881 |
| 249 | ·751 | 1 | 1 | 1·8441 | 204 | ·796 | 0 | 17 | 3·9687 |
| 248 | ·752 | 1 | 1 | 0·8247 | 203 | ·797 | 0 | 17 | 2·9492 |
| 247 | ·753 | 1 | 0 | 11·8052 | 202 | ·798 | 0 | 17 | 1·9298 |
| 246 | ·754 | 1 | 0 | 10·7858 | 201 | ·799 | 0 | 17 | 0·9103 |
| 245 | ·755 | 1 | 0 | 9·7663 | 200 | ·800 | 0 | 16 | 11·8909 |
| 244 | ·756 | 1 | 0 | 8·7469 | 199 | ·801 | 0 | 16 | 10·8714 |
| 243 | ·757 | 1 | 0 | 7·7274 | 198 | ·802 | 0 | 16 | 9·8519 |
| 242 | ·758 | 1 | 0 | 6·7079 | 197 | ·803 | 0 | 16 | 8·8325 |
| 241 | ·759 | 1 | 0 | 5·6885 | 196 | ·804 | 0 | 16 | 7·8130 |
| 240 | ·760 | 1 | 0 | 4·6690 | 195 | ·805 | 0 | 16 | 6·7936 |
| 239 | ·761 | 1 | 0 | 3·6496 | 194 | ·806 | 0 | 16 | 5·7741 |
| 238 | ·762 | 1 | 0 | 2·6301 | 193 | ·807 | 0 | 16 | 4·7547 |
| 237 | ·763 | 1 | 0 | 1·6107 | 192 | ·808 | 0 | 16 | 3·7352 |
| 236 | ·764 | 1 | 0 | 0·5912 | 191 | ·809 | 0 | 16 | 2·7158 |
| 235 | ·765 | 0 | 19 | 11·5718 | 190 | ·810 | 0 | 16 | 1·6963 |
| 234 | ·766 | 0 | 19 | 10·5523 | 189 | ·811 | 0 | 16 | 0·6769 |
| 233 | ·767 | 0 | 19 | 9·5329 | 188 | ·812 | 0 | 15 | 11·6574 |
| 232 | ·768 | 0 | 19 | 8·5134 | 187 | ·813 | 0 | 15 | 10·6379 |
| 231 | ·769 | 0 | 19 | 7·4939 | 186 | ·814 | 0 | 15 | 9·6185 |
| 230 | ·770 | 0 | 19 | 6·4745 | 185 | ·815 | 0 | 15 | 8·5990 |
| 229 | ·771 | 0 | 19 | 5·4551 | 184 | ·816 | 0 | 15 | 7·5796 |
| 228 | ·772 | 0 | 19 | 4·4356 | 183 | ·817 | 0 | 15 | 6·5601 |
| 227 | ·773 | 0 | 19 | 3·4161 | 182 | ·818 | 0 | 15 | 5·5407 |
| 226 | ·774 | 0 | 19 | 2·3967 | 181 | ·819 | 0 | 15 | 4·5212 |
| 225 | ·775 | 0 | 19 | 1·3772 | 180 | ·820 | 0 | 15 | 3·5018 |
| 224 | ·776 | 0 | 19 | 0·3578 | 179 | ·821 | 0 | 15 | 2·4823 |
| 223 | ·777 | 0 | 18 | 11·3383 | 178 | ·822 | 0 | 15 | 1·4629 |
| 222 | ·778 | 0 | 18 | 10·3189 | 177 | ·823 | 0 | 15 | 0·4434 |
| 221 | ·779 | 0 | 18 | 9·2994 | 176 | ·824 | 0 | 14 | 11·4239 |
| 220 | ·780 | 0 | 18 | 8·2799 | 175 | ·825 | 0 | 14 | 10·4045 |
| 219 | ·781 | 0 | 18 | 7·2605 | 174 | ·826 | 0 | 14 | 9·3851 |
| 218 | ·782 | 0 | 18 | 6·2410 | 173 | ·827 | 0 | 14 | 8·3656 |
| 217 | ·783 | 0 | 18 | 5·2216 | 172 | ·828 | 0 | 14 | 7·3461 |
| 216 | ·784 | 0 | 18 | 4·2021 | 171 | ·829 | 0 | 14 | 6·3267 |
| 215 | ·785 | 0 | 18 | 3·1827 | 170 | ·830 | 0 | 14 | 5·3072 |
| 214 | ·786 | 0 | 18 | 2·1632 | 169 | ·831 | 0 | 14 | 4·2878 |
| 213 | ·787 | 0 | 18 | 1·1438 | 168 | ·832 | 0 | 14 | 3·2683 |
| 212 | ·788 | 0 | 18 | 0·1243 | 167 | ·833 | 0 | 14 | 2·2489 |

| Fine Gold | Alloy | Value | | | Fine Gold | Alloy | Value | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 166 | ·834 | 0 | 14 | 1·2294 | 121 | ·879 | 0 | 10 | 3·3530 |
| 165 | ·835 | 0 | 14 | 0·2099 | 120 | ·880 | 0 | 10 | 2·3345 |
| 164 | ·836 | 0 | 13 | 11·1905 | 119 | ·881 | 0 | 10 | 1·3151 |
| 163 | ·837 | 0 | 13 | 10·1710 | 118 | ·882 | 0 | 10 | 0·2956 |
| 162 | ·838 | 0 | 13 | 9·1516 | 117 | ·883 | 0 | 9 | 11·2761 |
| 161 | ·839 | 0 | 13 | 8·1321 | 116 | ·884 | 0 | 9 | 10·2567 |
| 160 | ·840 | 0 | 13 | 7·1127 | 115 | ·885 | 0 | 9 | 9·2372 |
| 159 | ·841 | 0 | 13 | 6·0932 | 114 | ·886 | 0 | 9 | 8·2178 |
| 158 | ·842 | 0 | 13 | 5·0738 | 113 | ·887 | 0 | 9 | 7·1983 |
| 157 | ·843 | 0 | 13 | 4·0543 | 112 | ·888 | 0 | 9 | 6·1789 |
| 156 | ·844 | 0 | 13 | 3·0349 | 111 | ·889 | 0 | 9 | 5·1594 |
| 155 | ·845 | 0 | 13 | 2·0154 | 110 | ·890 | 0 | 9 | 4·1399 |
| 154 | ·846 | 0 | 13 | 0·9959 | 109 | ·891 | 0 | 9 | 3·1205 |
| 153 | ·847 | 0 | 12 | 11·9765 | 108 | ·892 | 0 | 9 | 2·1010 |
| 152 | ·848 | 0 | 12 | 10·9570 | 107 | ·893 | 0 | 9 | 1·0816 |
| 151 | ·849 | 0 | 12 | 9·9376 | 106 | ·894 | 0 | 9 | 0·0621 |
| 150 | ·850 | 0 | 12 | 8·9181 | 105 | ·895 | 0 | 8 | 11·0427 |
| 149 | ·851 | 0 | 12 | 7·8987 | 104 | ·896 | 0 | 8 | 10·0232 |
| 148 | ·852 | 0 | 12 | 6·8792 | 103 | ·897 | 0 | 8 | 9·0038 |
| 147 | ·853 | 0 | 12 | 5·8598 | 102 | ·898 | 0 | 8 | 7·9843 |
| 146 | ·854 | 0 | 12 | 4·8403 | 101 | ·899 | 0 | 8 | 6·9649 |
| 145 | ·855 | 0 | 12 | 3·8209 | 100 | ·900 | 0 | 8 | 5·9454 |
| 144 | ·856 | 0 | 12 | 2·8014 | 99 | ·901 | 0 | 8 | 4·9259 |
| 143 | ·857 | 0 | 12 | 1·7819 | 98 | ·902 | 0 | 8 | 3·9065 |
| 142 | ·858 | 0 | 12 | 0·7625 | 97 | ·903 | 0 | 8 | 2·8870 |
| 141 | ·859 | 0 | 11 | 11·7430 | 96 | ·904 | 0 | 8 | 1·8676 |
| 140 | ·860 | 0 | 11 | 10·7236 | 95 | ·905 | 0 | 8 | 0·8481 |
| 139 | ·861 | 0 | 11 | 9·7041 | 94 | ·906 | 0 | 7 | 11·8287 |
| 138 | ·862 | 0 | 11 | 8·6847 | 93 | ·907 | 0 | 7 | 10·8092 |
| 137 | ·863 | 0 | 11 | 7·6652 | 92 | ·908 | 0 | 7 | 8·7898 |
| 136 | ·864 | 0 | 11 | 6·6458 | 91 | ·909 | 0 | 7 | 9·7703 |
| 135 | ·865 | 0 | 11 | 5·6263 | 90 | ·910 | 0 | 7 | 7·7509 |
| 134 | ·866 | 0 | 11 | 4·6069 | 89 | ·911 | 0 | 7 | 6·7314 |
| 133 | ·867 | 0 | 11 | 3·5874 | 88 | ·912 | 0 | 7 | 5·7119 |
| 132 | ·868 | 0 | 11 | 2·5679 | 87 | ·913 | 0 | 7 | 4·6925 |
| 131 | ·869 | 0 | 11 | 1·5485 | 86 | ·914 | 0 | 7 | 3·6730 |
| 130 | ·870 | 0 | 11 | 0·5290 | 85 | ·915 | 0 | 7 | 2·6536 |
| 129 | ·871 | 0 | 10 | 11·5096 | 84 | ·916 | 0 | 7 | 1·6341 |
| 128 | ·872 | 0 | 10 | 10·4901 | 83 | ·917 | 0 | 7 | 10·6147 |
| 127 | ·873 | 0 | 10 | 9·4707 | 82 | ·918 | 0 | 6 | 11·5952 |
| 126 | ·874 | 0 | 10 | 8·4512 | 81 | ·919 | 0 | 6 | 0·5758 |
| 125 | ·875 | 0 | 10 | 7·4318 | 80 | ·920 | 0 | 6 | 9·5563 |
| 124 | ·876 | 0 | 10 | 6·4123 | 79 | ·921 | 0 | 6 | 8·5369 |
| 123 | ·877 | 0 | 10 | 5·3929 | 78 | ·922 | 0 | 6 | 7·5174 |
| 122 | ·878 | 0 | 10 | 4·3734 | 77 | ·923 | 0 | 6 | 6·4979 |

| Fine Gold | Alloy | Value | | | Fine Gold | Alloy | Value | | |
|---|---|---|---|---|---|---|---|---|---|
| | | £ | s. | d. | | | £ | s. | d. |
| 76 | ·924 | 0 | 6 | 5·4785 | 38 | ·962 | 0 | 3 | 2·7392 |
| 75 | ·925 | 0 | 6 | 4·4590 | 37 | ·963 | 0 | 3 | 1·7198 |
| 74 | ·926 | 0 | 6 | 3·4396 | 36 | ·964 | 0 | 3 | 0·7003 |
| 73 | ·927 | 0 | 6 | 2·4201 | 35 | ·965 | 0 | 2 | 11·6809 |
| 72 | ·928 | 0 | 6 | 1·4007 | 34 | ·966 | 0 | 2 | 10·6614 |
| 71 | ·929 | 0 | 6 | 0·3812 | 33 | ·967 | 0 | 2 | 9·6419 |
| 70 | ·930 | 0 | 5 | 11·3618 | 32 | ·968 | 0 | 2 | 8·6225 |
| 69 | ·931 | 0 | 5 | 10·3423 | 31 | ·969 | 0 | 2 | 7·6030 |
| 68 | ·932 | 0 | 5 | 9·3229 | 30 | ·970 | 0 | 2 | 6·5836 |
| 67 | ·933 | 0 | 5 | 8·3034 | 29 | ·971 | 0 | 2 | 5·5641 |
| 66 | ·934 | 0 | 5 | 7·2839 | 28 | ·972 | 0 | 2 | 4·5447 |
| 65 | ·935 | 0 | 5 | 6·2645 | 27 | ·973 | 0 | 2 | 3·5252 |
| 64 | ·936 | 0 | 5 | 5·2451 | 26 | ·974 | 0 | 2 | 2·5058 |
| 63 | ·937 | 0 | 5 | 4·2256 | 25 | ·975 | 0 | 2 | 1·4863 |
| 62 | ·938 | 0 | 5 | 3·2061 | 24 | ·976 | 0 | 2 | 0·4669 |
| 61 | ·939 | 0 | 5 | 2·1867 | 23 | ·977 | 0 | 1 | 11·4474 |
| 60 | ·940 | 0 | 5 | 1·1672 | 22 | ·978 | 0 | 1 | 10·4279 |
| 59 | ·941 | 0 | 5 | 0·1478 | 21 | ·979 | 0 | 1 | 9·4085 |
| 58 | ·942 | 0 | 4 | 11·1283 | 20 | ·980 | 0 | 1 | 8·3890 |
| 57 | ·943 | 0 | 4 | 10·1089 | 19 | ·981 | 0 | 1 | 7·3696 |
| 56 | ·944 | 0 | 4 | 9·0894 | 18 | ·982 | 0 | 1 | 6·3501 |
| 55 | ·945 | 0 | 4 | 8·0699 | 17 | ·983 | 0 | 1 | 5·3307 |
| 54 | ·946 | 0 | 4 | 7·0505 | 16 | ·984 | 0 | 1 | 4·3112 |
| 53 | ·947 | 0 | 4 | 6·0310 | 15 | ·985 | 0 | 1 | 3·2918 |
| 52 | ·948 | 0 | 4 | 5·0116 | 14 | ·986 | 0 | 1 | 2·2723 |
| 51 | ·949 | 0 | 4 | 3·9921 | 13 | ·987 | 0 | 1 | 1·2529 |
| 50 | ·950 | 0 | 4 | 2·9727 | 12 | ·988 | 0 | 1 | 0·2334 |
| 49 | ·951 | 0 | 4 | 1·9532 | 11 | ·989 | 0 | 0 | 11·2139 |
| 48 | ·952 | 0 | 4 | 0·9338 | 10 | ·990 | 0 | 0 | 10·1945 |
| 47 | ·953 | 0 | 3 | 11·9143 | 9 | ·991 | 0 | 0 | 9·1750 |
| 46 | ·954 | 0 | 3 | 10·8949 | 8 | ·992 | 0 | 0 | 8·1556 |
| 45 | ·955 | 0 | 3 | 9·8754 | 7 | ·993 | 0 | 0 | 7·1361 |
| 44 | ·956 | 0 | 3 | 8·8559 | 6 | ·994 | 0 | 0 | 6·1167 |
| 43 | ·957 | 0 | 3 | 7·8365 | 5 | ·995 | 0 | 0 | 5·0972 |
| 42 | ·958 | 0 | 3 | 6·8170 | 4 | ·996 | 0 | 0 | 4·0778 |
| 41 | ·959 | 0 | 3 | 5·7976 | 3 | ·997 | 0 | 0 | 3·0583 |
| 40 | ·960 | 0 | 3 | 4·7781 | 2 | ·998 | 0 | 0 | 2·0389 |
| 39 | ·961 | 0 | 3 | 3·7587 | 1 | ·999 | 0 | 0 | 1·0194 |

### To convert MINT VALUE into BANK VALUE when the Standard is expressed in Thousandths.

| Thousandths. | Value in Pence. | Thousandths. | Value in Pence. |
|---|---|---|---|
| 1 | ·001636 | 6 | ·009816 |
| 2 | ·003272 | 7 | ·011352 |
| 3 | ·004908 | 8 | ·013088 |
| 4 | ·006544 | 9 | ·014724 |
| 5 | ·008180 | | |

To illustrate the use of the above table, gold of $\frac{500}{1000}$ths fine may be taken. As in the Table for finding the Bank value of gold when the standard is reported in carats, &c., the amounts in pence, as above, are to be deducted from the prices attached to corresponding standards in Table No. 2. Thus, the minus value of $\frac{5}{1000}$ths is ·00818 of a penny; therefore, the minus value of $\frac{500}{1000}$th is ·818 of a penny, which amount must be deducted from the Mint price of gold at the above standard. On referring to the Table it will be found to be £2 2*s.* 5·7272*d.* per oz. Now, if ·818 be deducted, the remainder will be £2 2*s.* 4·9092*d.*, representing the Bank value of 1 oz. of gold of the fineness just mentioned.

## TABLE III.

**Assay Table**, showing the Amount of **Gold** or **Silver**, in Ounces, Pennyweights, and Grains, contained in a Ton of Ore, &c. from the Weight of Metal obtained in an Assay of 200 Grains of Mineral.

| If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | |
|---|---|---|---|
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·001 | 0 | 3 | 6 |
| ·002 | 0 | 6 | 12 |
| ·003 | 0 | 9 | 19 |
| ·004 | 0 | 13 | 1 |
| ·005 | 0 | 16 | 8 |
| ·006 | 0 | 19 | 14 |
| ·007 | 1 | 2 | 20 |
| ·008 | 1 | 6 | 3 |
| ·009 | 1 | 9 | 9 |
| ·010 | 1 | 12 | 6 |
| ·011 | 1 | 15 | 22 |
| ·012 | 1 | 19 | 4 |
| ·013 | 2 | 2 | 11 |
| ·014 | 2 | 5 | 17 |
| ·015 | 2 | 9 | 0 |
| ·016 | 2 | 12 | 6 |
| ·017 | 2 | 15 | 12 |
| ·018 | 2 | 18 | 19 |
| ·019 | 3 | 2 | 1 |
| ·020 | 3 | 5 | 8 |
| ·021 | 3 | 8 | 14 |
| ·022 | 3 | 11 | 20 |
| ·023 | 3 | 15 | 3 |
| ·024 | 3 | 18 | 9 |
| ·025 | 4 | 1 | 16 |
| ·026 | 4 | 4 | 22 |
| ·027 | 4 | 8 | 4 |
| ·028 | 4 | 11 | 11 |
| ·029 | 4 | 14 | 17 |
| ·030 | 4 | 18 | 0 |
| ·031 | 5 | 1 | 6 |
| ·032 | 5 | 4 | 12 |
| ·033 | 5 | 7 | 19 |
| ·034 | 5 | 11 | 1 |
| ·035 | 5 | 14 | 8 |
| ·036 | 5 | 17 | 14 |
| ·037 | 6 | 0 | 20 |
| ·038 | 6 | 4 | 3 |
| ·039 | 6 | 7 | 9 |
| ·040 | 6 | 10 | 16 |
| ·041 | 6 | 13 | 22 |
| ·042 | 6 | 17 | 4 |
| ·043 | 7 | 0 | 11 |
| ·044 | 7 | 3 | 17 |
| ·045 | 7 | 7 | 0 |
| ·046 | 7 | 10 | 6 |
| ·047 | 7 | 13 | 12 |
| ·048 | 7 | 16 | 19 |
| ·049 | 8 | 0 | 1 |
| ·050 | 8 | 3 | 8 |
| ·051 | 8 | 6 | 14 |
| ·052 | 8 | 9 | 20 |
| ·053 | 8 | 13 | 3 |
| ·054 | 8 | 16 | 9 |
| ·055 | 8 | 19 | 16 |
| ·056 | 9 | 2 | 22 |
| ·057 | 9 | 6 | 4 |
| ·058 | 9 | 9 | 11 |
| ·059 | 9 | 12 | 17 |
| ·060 | 9 | 16 | 0 |

| If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | |
|---|---|---|---|
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·061 | 9 | 19 | 6 |
| ·062 | 10 | 2 | 12 |
| ·063 | 10 | 5 | 19 |
| ·064 | 10 | 9 | 1 |
| ·065 | 10 | 12 | 8 |
| ·066 | 10 | 15 | 14 |
| ·067 | 10 | 18 | 20 |
| ·068 | 11 | 2 | 3 |
| ·069 | 11 | 5 | 9 |
| ·070 | 11 | 8 | 16 |
| ·071 | 11 | 11 | 22 |
| ·072 | 11 | 15 | 4 |
| ·073 | 11 | 18 | 11 |
| ·074 | 12 | 1 | 17 |
| ·075 | 12 | 5 | 0 |
| ·076 | 12 | 8 | 6 |
| ·077 | 12 | 11 | 12 |
| ·078 | 12 | 14 | 19 |
| ·079 | 12 | 18 | 1 |
| ·080 | 13 | 1 | 8 |
| ·081 | 13 | 4 | 14 |
| ·082 | 13 | 7 | 20 |
| ·083 | 13 | 11 | 3 |
| ·084 | 13 | 14 | 9 |
| ·085 | 13 | 17 | 16 |
| ·086 | 14 | 0 | 22 |
| ·087 | 14 | 4 | 4 |
| ·088 | 14 | 7 | 11 |
| ·089 | 14 | 10 | 17 |
| ·090 | 14 | 14 | 0 |
| ·091 | 14 | 17 | 6 |
| ·092 | 15 | 0 | 12 |
| ·093 | 15 | 3 | 19 |
| ·094 | 15 | 7 | 1 |
| ·095 | 15 | 10 | 8 |
| ·096 | 15 | 13 | 14 |
| ·097 | 15 | 16 | 20 |
| ·098 | 16 | 0 | 3 |
| ·099 | 16 | 3 | 9 |
| ·100 | 16 | 6 | 16 |
| ·101 | 16 | 9 | 22 |
| ·102 | 16 | 13 | 4 |
| ·103 | 16 | 16 | 11 |
| ·104 | 16 | 19 | 17 |
| ·105 | 17 | 3 | 0 |
| ·106 | 17 | 6 | 6 |
| ·107 | 17 | 9 | 12 |
| ·108 | 17 | 12 | 19 |
| ·109 | 17 | 16 | 1 |
| ·110 | 17 | 19 | 8 |
| ·111 | 18 | 2 | 14 |
| ·112 | 18 | 5 | 20 |
| ·113 | 18 | 9 | 3 |
| ·114 | 18 | 12 | 9 |
| ·115 | 18 | 15 | 16 |
| ·116 | 18 | 18 | 22 |
| ·117 | 19 | 2 | 4 |
| ·118 | 19 | 5 | 11 |
| ·119 | 19 | 8 | 17 |
| ·120 | 19 | 12 | 0 |
| ·121 | 19 | 15 | 6 |
| ·122 | 19 | 18 | 12 |
| ·123 | 20 | 1 | 19 |
| ·124 | 20 | 5 | 1 |
| ·125 | 20 | 8 | 8 |
| ·126 | 20 | 11 | 14 |
| ·127 | 20 | 14 | 20 |
| ·128 | 20 | 18 | 3 |
| ·129 | 21 | 1 | 9 |
| ·130 | 21 | 4 | 16 |
| ·131 | 21 | 7 | 22 |
| ·132 | 21 | 11 | 4 |
| ·133 | 21 | 14 | 11 |
| ·134 | 21 | 17 | 17 |
| ·135 | 22 | 1 | 0 |
| ·136 | 22 | 4 | 6 |
| ·137 | 22 | 7 | 12 |
| ·138 | 22 | 10 | 19 |
| ·139 | 22 | 14 | 1 |
| ·140 | 22 | 17 | 8 |
| ·141 | 23 | 0 | 14 |
| ·142 | 23 | 3 | 20 |
| ·143 | 23 | 7 | 3 |
| ·144 | 23 | 10 | 9 |
| ·145 | 23 | 13 | 16 |
| ·146 | 23 | 16 | 22 |
| ·147 | 24 | 0 | 4 |
| ·148 | 24 | 3 | 11 |

| If 200 grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | | If 200 grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | |
|---|---|---|---|---|---|---|---|
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* | *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·149 | 24 | 6 | 17 | ·193 | 31 | 10 | 11 |
| ·150 | 24 | 10 | 0 | ·194 | 31 | 13 | 17 |
| ·151 | 24 | 13 | 6 | ·195 | 31 | 17 | 0 |
| ·152 | 24 | 16 | 12 | ·196 | 32 | 0 | 6 |
| ·153 | 24 | 19 | 19 | ·197 | 32 | 3 | 12 |
| ·154 | 25 | 3 | 1 | ·198 | 32 | 6 | 19 |
| ·155 | 25 | 6 | 8 | ·199 | 32 | 10 | 1 |
| ·156 | 25 | 9 | 14 | ·200 | 32 | 13 | 8 |
| ·157 | 25 | 12 | 20 | ·201 | 32 | 16 | 14 |
| ·158 | 25 | 16 | 3 | ·202 | 32 | 19 | 20 |
| ·159 | 25 | 19 | 9 | ·203 | 33 | 3 | 3 |
| ·160 | 26 | 2 | 16 | ·204 | 33 | 6 | 9 |
| ·161 | 26 | 5 | 22 | ·205 | 33 | 9 | 16 |
| ·162 | 26 | 9 | 4 | ·206 | 33 | 12 | 22 |
| ·163 | 26 | 12 | 11 | ·207 | 33 | 16 | 4 |
| ·164 | 26 | 15 | 17 | ·208 | 33 | 19 | 11 |
| ·165 | 26 | 19 | 0 | ·209 | 34 | 2 | 17 |
| ·166 | 27 | 2 | 6 | ·210 | 34 | 6 | 0 |
| ·167 | 27 | 5 | 12 | ·211 | 34 | 9 | 6 |
| ·168 | 27 | 8 | 19 | ·212 | 34 | 12 | 12 |
| ·169 | 27 | 12 | 1 | ·213 | 34 | 15 | 19 |
| ·170 | 27 | 15 | 8 | ·214 | 34 | 19 | 1 |
| ·171 | 27 | 18 | 14 | ·215 | 35 | 2 | 8 |
| ·172 | 28 | 1 | 20 | ·216 | 35 | 5 | 14 |
| ·173 | 28 | 5 | 3 | ·217 | 35 | 8 | 20 |
| ·174 | 28 | 8 | 9 | ·218 | 35 | 12 | 3 |
| ·175 | 28 | 11 | 16 | ·219 | 35 | 15 | 9 |
| ·176 | 28 | 14 | 22 | ·220 | 35 | 18 | 16 |
| ·177 | 28 | 18 | 4 | ·221 | 36 | 1 | 22 |
| ·178 | 29 | 1 | 11 | ·222 | 36 | 5 | 4 |
| ·179 | 29 | 4 | 17 | ·223 | 36 | 8 | 11 |
| ·180 | 29 | 8 | 0 | ·224 | 36 | 11 | 17 |
| ·181 | 29 | 11 | 6 | ·225 | 36 | 15 | 0 |
| ·182 | 29 | 14 | 12 | ·226 | 36 | 18 | 6 |
| ·183 | 29 | 17 | 19 | ·227 | 37 | 1 | 12 |
| ·184 | 30 | 1 | 1 | ·228 | 37 | 4 | 19 |
| ·185 | 30 | 4 | 8 | ·229 | 37 | 8 | 1 |
| ·186 | 30 | 7 | 14 | ·230 | 37 | 11 | 8 |
| ·187 | 30 | 10 | 20 | ·231 | 37 | 14 | 14 |
| ·188 | 30 | 14 | 3 | ·232 | 37 | 17 | 20 |
| ·189 | 30 | 17 | 9 | ·233 | 38 | 1 | 3 |
| ·190 | 31 | 0 | 16 | ·234 | 38 | 4 | 9 |
| ·191 | 31 | 3 | 22 | ·235 | 38 | 7 | 16 |
| ·192 | 31 | 7 | 4 | ·236 | 38 | 10 | 22 |

| If 200 Grains of Ore give of | One Ton of Ore will yield of | | | If 200 Grains of Ore give of | One Ton of Ore will yield of | | |
|---|---|---|---|---|---|---|---|
| FINE METAL | FINE METAL | | | FINE METAL | FINE METAL | | |
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* | *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·237 | 38 | 14 | 4 | ·281 | 45 | 17 | 22 |
| ·238 | 38 | 17 | 11 | ·282 | 46 | 1 | 4 |
| ·239 | 39 | 0 | 17 | ·283 | 46 | 4 | 11 |
| ·240 | 39 | 4 | 0 | ·284 | 46 | 7 | 17 |
| ·241 | 39 | 7 | 6 | ·285 | 46 | 11 | 0 |
| ·242 | 39 | 10 | 12 | ·286 | 46 | 14 | 6 |
| ·243 | 39 | 13 | 18 | ·287 | 46 | 17 | 12 |
| ·244 | 39 | 17 | 1 | ·288 | 47 | 0 | 19 |
| ·245 | 40 | 0 | 8 | ·289 | 47 | 4 | 1 |
| ·246 | 40 | 3 | 14 | ·290 | 47 | 7 | 8 |
| ·247 | 40 | 6 | 20 | ·291 | 47 | 10 | 14 |
| ·248 | 40 | 10 | 3 | ·292 | 47 | 13 | 20 |
| ·249 | 40 | 13 | 9 | ·293 | 47 | 17 | 3 |
| ·250 | 40 | 16 | 16 | ·294 | 48 | 0 | 9 |
| ·251 | 40 | 19 | 22 | ·295 | 48 | 3 | 16 |
| ·252 | 41 | 3 | 4 | ·296 | 48 | 6 | 22 |
| ·253 | 41 | 6 | 11 | ·297 | 48 | 10 | 4 |
| ·254 | 41 | 9 | 17 | ·298 | 48 | 13 | 11 |
| ·255 | 41 | 13 | 0 | ·299 | 48 | 16 | 17 |
| ·256 | 41 | 16 | 6 | ·300 | 49 | 0 | 0 |
| ·257 | 41 | 19 | 12 | ·301 | 49 | 3 | 6 |
| ·258 | 42 | 2 | 19 | ·302 | 49 | 6 | 12 |
| ·259 | 42 | 6 | 1 | ·303 | 49 | 9 | 19 |
| ·260 | 42 | 9 | 8 | ·304 | 49 | 13 | 1 |
| ·261 | 42 | 12 | 14 | ·305 | 49 | 16 | 8 |
| ·262 | 42 | 15 | 20 | ·306 | 49 | 19 | 14 |
| ·263 | 42 | 19 | 3 | ·307 | 50 | 2 | 20 |
| ·264 | 43 | 2 | 9 | ·308 | 50 | 6 | 3 |
| ·265 | 43 | 5 | 16 | ·309 | 50 | 9 | 9 |
| ·266 | 43 | 8 | 22 | ·310 | 50 | 12 | 16 |
| ·267 | 43 | 12 | 4 | ·311 | 50 | 15 | 22 |
| ·268 | 43 | 15 | 11 | ·312 | 50 | 19 | 4 |
| ·269 | 43 | 18 | 17 | ·313 | 51 | 2 | 11 |
| ·270 | 44 | 2 | 0 | ·314 | 51 | 5 | 17 |
| ·271 | 44 | 5 | 6 | ·315 | 51 | 9 | 0 |
| ·272 | 44 | 8 | 12 | ·316 | 51 | 12 | 6 |
| ·273 | 44 | 11 | 19 | ·317 | 51 | 15 | 12 |
| ·274 | 44 | 15 | 1 | ·318 | 51 | 18 | 19 |
| ·275 | 44 | 18 | 8 | ·319 | 52 | 2 | 1 |
| ·276 | 45 | 1 | 14 | ·320 | 52 | 5 | 8 |
| ·277 | 45 | 4 | 20 | ·321 | 52 | 8 | 14 |
| ·278 | 45 | 8 | 3 | ·322 | 52 | 11 | 20 |
| ·279 | 45 | 11 | 9 | ·323 | 52 | 15 | 3 |
| ·280 | 45 | 14 | 16 | ·324 | 52 | 18 | 9 |

| If 200 Grains of Ore give of | One Ton of Ore will yield of | | | If 200 Grains of Ore give of | One Ton of Ore will yield of | | |
|---|---|---|---|---|---|---|---|
| FINE METAL | FINE METAL | | | FINE METAL | FINE METAL | | |
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* | *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·325 | 53 | 1 | 16 | ·369 | 60 | 5 | 9 |
| ·326 | 53 | 4 | 22 | ·370 | 60 | 8 | 16 |
| ·327 | 53 | 8 | 4 | ·371 | 60 | 11 | 22 |
| ·328 | 53 | 11 | 11 | ·372 | 60 | 15 | 4 |
| ·329 | 53 | 14 | 17 | ·373 | 60 | 18 | 11 |
| ·330 | 53 | 18 | 0 | ·374 | 61 | 1 | 17 |
| ·331 | 54 | 1 | 6 | ·375 | 61 | 5 | 0 |
| ·332 | 54 | 4 | 12 | ·376 | 61 | 8 | 6 |
| ·333 | 54 | 7 | 19 | ·377 | 61 | 11 | 12 |
| ·334 | 54 | 11 | 1 | ·378 | 61 | 14 | 19 |
| ·335 | 54 | 14 | 8 | ·379 | 61 | 18 | 1 |
| ·336 | 54 | 17 | 14 | ·380 | 62 | 1 | 8 |
| ·337 | 55 | 0 | 20 | ·381 | 62 | 4 | 14 |
| ·338 | 55 | 4 | 3 | ·382 | 62 | 7 | 20 |
| ·339 | 55 | 7 | 9 | ·383 | 62 | 11 | 3 |
| ·340 | 55 | 10 | 16 | ·384 | 62 | 14 | 9 |
| ·341 | 55 | 13 | 22 | ·385 | 62 | 17 | 16 |
| ·342 | 55 | 17 | 4 | ·386 | 63 | 0 | 22 |
| ·343 | 56 | 0 | 11 | ·387 | 63 | 4 | 4 |
| ·344 | 56 | 3 | 17 | ·388 | 63 | 7 | 11 |
| ·345 | 56 | 7 | 0 | ·389 | 63 | 10 | 17 |
| ·346 | 56 | 10 | 6 | ·390 | 63 | 14 | 0 |
| ·347 | 56 | 13 | 12 | ·391 | 63 | 17 | 6 |
| ·348 | 56 | 16 | 19 | ·392 | 64 | 0 | 12 |
| ·349 | 57 | 0 | 1 | ·393 | 64 | 3 | 19 |
| ·350 | 57 | 3 | 8 | ·394 | 64 | 7 | 1 |
| ·351 | 57 | 6 | 14 | ·395 | 64 | 10 | 8 |
| ·352 | 57 | 9 | 20 | ·396 | 64 | 13 | 14 |
| ·353 | 57 | 13 | 3 | ·397 | 64 | 16 | 20 |
| ·354 | 57 | 16 | 9 | ·398 | 65 | 0 | 3 |
| ·355 | 57 | 19 | 16 | ·399 | 65 | 3 | 9 |
| ·356 | 58 | 2 | 22 | ·400 | 65 | 6 | 16 |
| ·357 | 58 | 6 | 4 | ·401 | 65 | 9 | 22 |
| ·358 | 58 | 9 | 11 | ·402 | 65 | 13 | 4 |
| ·359 | 58 | 12 | 17 | ·403 | 65 | 16 | 11 |
| ·360 | 58 | 16 | 0 | ·404 | 65 | 19 | 17 |
| ·361 | 58 | 19 | 6 | ·405 | 66 | 3 | 0 |
| ·362 | 59 | 9 | 12 | ·406 | 66 | 6 | 6 |
| ·363 | 59 | 5 | 19 | ·407 | 66 | 9 | 12 |
| ·364 | 59 | 9 | 1 | ·408 | 66 | 12 | 19 |
| ·365 | 59 | 12 | 8 | ·409 | 66 | 16 | 1 |
| ·366 | 59 | 15 | 14 | ·410 | 66 | 19 | 8 |
| ·367 | 59 | 18 | 20 | ·411 | 67 | 2 | 14 |
| ·368 | 60 | 2 | 3 | ·412 | 67 | 5 | 20 |

| If 200 Grains of Ore give of | One Ton of Ore will yield of | | |
|---|---|---|---|
| FINE METAL | FINE METAL | | |
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·413 | 67 | 9 | 3 |
| ·414 | 67 | 12 | 9 |
| ·415 | 67 | 15 | 16 |
| ·416 | 67 | 18 | 22 |
| ·417 | 68 | 2 | 4 |
| ·418 | 68 | 5 | 11 |
| ·419 | 68 | 8 | 17 |
| ·420 | 68 | 12 | 0 |
| ·421 | 68 | 15 | 6 |
| ·422 | 68 | 18 | 12 |
| ·423 | 69 | 1 | 19 |
| ·424 | 69 | 5 | 1 |
| ·425 | 69 | 8 | 8 |
| ·426 | 69 | 11 | 14 |
| ·427 | 69 | 14 | 20 |
| ·428 | 69 | 18 | 3 |
| ·429 | 70 | 1 | 9 |
| ·430 | 70 | 4 | 16 |
| ·431 | 70 | 7 | 22 |
| ·432 | 70 | 11 | 4 |
| ·433 | 70 | 14 | 11 |
| ·434 | 70 | 17 | 17 |
| ·435 | 71 | 1 | 0 |
| ·436 | 71 | 4 | 6 |
| ·437 | 71 | 7 | 12 |
| ·438 | 71 | 10 | 19 |
| ·439 | 71 | 14 | 1 |
| ·440 | 71 | 17 | 8 |
| ·441 | 72 | 0 | 14 |
| ·442 | 72 | 3 | 20 |
| ·443 | 72 | 7 | 3 |
| ·444 | 72 | 10 | 9 |
| ·445 | 72 | 13 | 16 |
| ·446 | 72 | 16 | 22 |
| ·447 | 73 | 0 | 4 |
| ·448 | 73 | 3 | 11 |
| ·449 | 73 | 6 | 17 |
| ·450 | 73 | 10 | 0 |
| ·451 | 73 | 13 | 6 |
| ·452 | 73 | 16 | 12 |
| ·453 | 73 | 19 | 19 |
| ·454 | 74 | 3 | 1 |
| ·455 | 74 | 6 | 8 |
| ·456 | 74 | 9 | 14 |
| ·457 | 74 | 12 | 20 |
| ·458 | 74 | 16 | 3 |
| ·459 | 74 | 19 | 9 |
| ·460 | 75 | 2 | 16 |
| ·461 | 75 | 5 | 22 |
| ·462 | 75 | 9 | 4 |
| ·463 | 75 | 12 | 11 |
| ·464 | 75 | 15 | 17 |
| ·465 | 75 | 19 | 0 |
| ·466 | 76 | 2 | 6 |
| ·467 | 76 | 5 | 12 |
| ·468 | 76 | 8 | 19 |
| ·469 | 76 | 12 | 1 |
| ·470 | 76 | 15 | 8 |
| ·471 | 76 | 18 | 14 |
| ·472 | 77 | 1 | 20 |
| ·473 | 77 | 5 | 3 |
| ·474 | 77 | 8 | 9 |
| ·475 | 77 | 11 | 16 |
| ·476 | 77 | 14 | 22 |
| ·477 | 77 | 18 | 4 |
| ·478 | 78 | 1 | 11 |
| ·479 | 78 | 4 | 17 |
| ·480 | 78 | 8 | 0 |
| ·481 | 78 | 11 | 6 |
| ·482 | 78 | 14 | 12 |
| ·483 | 78 | 17 | 19 |
| ·484 | 79 | 1 | 1 |
| ·485 | 79 | 4 | 8 |
| ·486 | 79 | 7 | 14 |
| ·487 | 79 | 10 | 20 |
| ·488 | 79 | 14 | 3 |
| ·489 | 79 | 17 | 9 |
| ·490 | 80 | 0 | 16 |
| ·491 | 80 | 3 | 22 |
| ·492 | 80 | 7 | 4 |
| ·493 | 80 | 10 | 11 |
| ·494 | 80 | 13 | 17 |
| ·495 | 80 | 17 | 0 |
| ·496 | 81 | 0 | 6 |
| ·497 | 81 | 3 | 12 |
| ·498 | 81 | 6 | 19 |
| ·499 | 81 | 10 | 1 |
| ·500 | 81 | 13 | 8 |

| If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | | If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | |
|---|---|---|---|---|---|---|---|
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* | *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·501 | 81 | 16 | 14 | ·545 | 89 | 0 | 8 |
| ·502 | 81 | 19 | 20 | ·546 | 89 | 3 | 14 |
| ·503 | 82 | 3 | 3 | ·547 | 89 | 6 | 20 |
| ·504 | 82 | 6 | 9 | ·548 | 89 | 10 | 3 |
| ·505 | 82 | 9 | 16 | ·549 | 89 | 13 | 9 |
| ·506 | 82 | 12 | 22 | ·550 | 89 | 16 | 16 |
| ·507 | 82 | 16 | 4 | ·551 | 89 | 19 | 22 |
| ·508 | 82 | 19 | 11 | ·552 | 90 | 3 | 4 |
| ·509 | 83 | 2 | 17 | ·553 | 90 | 6 | 11 |
| ·510 | 83 | 6 | 0 | ·554 | 90 | 9 | 17 |
| ·511 | 83 | 9 | 6 | ·555 | 90 | 13 | 0 |
| ·512 | 83 | 12 | 12 | ·556 | 90 | 16 | 6 |
| ·513 | 83 | 15 | 19 | ·557 | 90 | 19 | 12 |
| ·514 | 83 | 19 | 1 | ·558 | 91 | 2 | 19 |
| ·515 | 84 | 2 | 8 | ·559 | 91 | 6 | 1 |
| ·516 | 84 | 5 | 14 | ·560 | 91 | 9 | 8 |
| ·517 | 84 | 8 | 20 | ·561 | 91 | 12 | 14 |
| ·518 | 84 | 12 | 3 | ·562 | 91 | 15 | 20 |
| ·519 | 84 | 15 | 9 | ·563 | 91 | 19 | 3 |
| ·520 | 84 | 18 | 16 | ·564 | 92 | 2 | 9 |
| ·521 | 85 | 1 | 22 | ·565 | 92 | 5 | 16 |
| ·522 | 85 | 5 | 4 | ·566 | 92 | 8 | 22 |
| ·523 | 85 | 8 | 11 | ·567 | 92 | 12 | 4 |
| ·524 | 85 | 11 | 17 | ·568 | 92 | 15 | 11 |
| ·525 | 85 | 15 | 0 | ·569 | 92 | 18 | 17 |
| ·526 | 85 | 18 | 6 | ·570 | 93 | 2 | 0 |
| ·527 | 86 | 1 | 12 | ·571 | 93 | 5 | 6 |
| ·528 | 86 | 4 | 19 | ·572 | 93 | 8 | 12 |
| ·529 | 86 | 8 | 1 | ·573 | 93 | 11 | 19 |
| ·530 | 86 | 11 | 8 | ·574 | 93 | 15 | 1 |
| ·531 | 86 | 14 | 14 | ·575 | 93 | 18 | 8 |
| ·532 | 86 | 17 | 20 | ·576 | 94 | 1 | 14 |
| ·533 | 87 | 1 | 3 | ·577 | 94 | 4 | 20 |
| ·534 | 87 | 4 | 9 | ·578 | 94 | 8 | 3 |
| ·535 | 87 | 7 | 16 | ·579 | 94 | 11 | 9 |
| ·536 | 87 | 10 | 22 | ·580 | 94 | 14 | 16 |
| ·537 | 87 | 14 | 4 | ·581 | 94 | 17 | 22 |
| ·538 | 87 | 17 | 11 | ·582 | 95 | 1 | 4 |
| ·539 | 88 | 0 | 17 | ·583 | 95 | 4 | 11 |
| ·540 | 88 | 4 | 0 | ·584 | 95 | 7 | 17 |
| ·541 | 88 | 7 | 6 | ·585 | 95 | 11 | 0 |
| ·542 | 88 | 10 | 12 | ·586 | 95 | 14 | 6 |
| ·543 | 88 | 13 | 19 | ·587 | 95 | 17 | 12 |
| ·544 | 88 | 17 | 1 | ·588 | 96 | 0 | 19 |

| If 200 Grains of Ore give of | One Ton of Ore will yield of | | | If 200 Grains of Ore give of | One Ton of Ore will yield of | | |
|---|---|---|---|---|---|---|---|
| FINE METAL | FINE METAL | | | FINE METAL | FINE METAL | | |
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* | *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·589 | 96 | 4 | 1 | ·633 | 103 | 7 | 19 |
| ·590 | 96 | 7 | 8 | ·634 | 103 | 11 | 1 |
| ·591 | 96 | 10 | 14 | ·635 | 103 | 14 | 8 |
| ·592 | 96 | 13 | 20 | ·636 | 103 | 17 | 14 |
| ·593 | 96 | 17 | 3 | ·637 | 104 | 0 | 20 |
| ·594 | 97 | 0 | 9 | ·638 | 104 | 4 | 3 |
| ·595 | 97 | 3 | 16 | ·639 | 104 | 7 | 9 |
| ·596 | 97 | 6 | 22 | ·640 | 104 | 10 | 16 |
| ·597 | 97 | 10 | 4 | ·641 | 104 | 13 | 22 |
| ·598 | 97 | 13 | 11 | ·642 | 104 | 17 | 4 |
| ·599 | 97 | 16 | 17 | ·643 | 105 | 0 | 11 |
| ·600 | 98 | 0 | 0 | ·644 | 105 | 3 | 17 |
| ·601 | 98 | 3 | 6 | ·645 | 105 | 7 | 0 |
| ·602 | 98 | 6 | 12 | ·646 | 105 | 10 | 6 |
| ·603 | 98 | 9 | 19 | ·647 | 105 | 13 | 12 |
| ·604 | 98 | 13 | 1 | ·648 | 105 | 16 | 19 |
| ·605 | 98 | 16 | 8 | ·649 | 106 | 0 | 1 |
| ·606 | 98 | 19 | 14 | ·650 | 106 | 3 | 8 |
| ·607 | 99 | 2 | 20 | ·651 | 106 | 6 | 14 |
| ·608 | 99 | 6 | 3 | ·652 | 106 | 9 | 20 |
| ·609 | 99 | 9 | 9 | ·653 | 106 | 13 | 3 |
| ·610 | 99 | 12 | 16 | ·654 | 106 | 16 | 9 |
| ·611 | 99 | 15 | 22 | ·655 | 106 | 19 | 16 |
| ·612 | 99 | 19 | 4 | ·656 | 107 | 2 | 22 |
| ·613 | 100 | 2 | 11 | ·657 | 107 | 6 | 4 |
| ·614 | 100 | 5 | 17 | ·658 | 107 | 9 | 11 |
| ·615 | 100 | 9 | 0 | ·659 | 107 | 12 | 17 |
| ·616 | 100 | 12 | 6 | ·660 | 107 | 16 | 0 |
| ·617 | 100 | 15 | 12 | ·661 | 107 | 19 | 6 |
| ·618 | 100 | 18 | 19 | ·662 | 108 | 2 | 12 |
| ·619 | 101 | 2 | 1 | ·663 | 108 | 5 | 19 |
| ·620 | 101 | 5 | 8 | ·664 | 108 | 9 | 1 |
| ·621 | 101 | 8 | 14 | ·665 | 108 | 12 | 8 |
| ·622 | 101 | 11 | 20 | ·666 | 108 | 15 | 14 |
| ·623 | 101 | 15 | 3 | ·667 | 108 | 18 | 20 |
| ·624 | 101 | 18 | 9 | ·668 | 109 | 2 | 3 |
| ·625 | 102 | 1 | 16 | ·669 | 109 | 5 | 9 |
| ·626 | 102 | 4 | 22 | ·670 | 109 | 8 | 16 |
| ·627 | 102 | 8 | 4 | ·671 | 109 | 11 | 22 |
| ·628 | 102 | 11 | 11 | ·672 | 109 | 15 | 4 |
| ·629 | 102 | 14 | 17 | ·673 | 109 | 18 | 11 |
| ·630 | 102 | 18 | 0 | ·674 | 110 | 1 | 17 |
| ·631 | 103 | 1 | 6 | ·675 | 110 | 5 | 0 |
| ·632 | 103 | 4 | 12 | ·676 | 110 | 8 | 6 |

| If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | | If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | |
|---|---|---|---|---|---|---|---|
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* | *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·677 | 110 | 11 | 12 | ·721 | 117 | 15 | 6 |
| ·678 | 110 | 14 | 19 | ·722 | 117 | 18 | 12 |
| ·679 | 110 | 18 | 1 | ·723 | 118 | 1 | 19 |
| ·680 | 111 | 1 | 8 | ·724 | 118 | 5 | 1 |
| ·681 | 111 | 4 | 14 | ·725 | 118 | 8 | 8 |
| ·682 | 111 | 7 | 20 | ·726 | 118 | 11 | 14 |
| ·683 | 111 | 11 | 3 | ·727 | 118 | 14 | 20 |
| ·684 | 111 | 14 | 9 | ·728 | 118 | 18 | 3 |
| ·685 | 111 | 17 | 6 | ·729 | 119 | 1 | 9 |
| ·686 | 112 | 0 | 22 | ·730 | 119 | 4 | 16 |
| ·687 | 112 | 4 | 4 | ·731 | 119 | 7 | 22 |
| ·688 | 112 | 7 | 11 | ·732 | 119 | 11 | 4 |
| ·689 | 112 | 10 | 17 | ·733 | 119 | 14 | 11 |
| ·690 | 112 | 14 | 0 | ·734 | 119 | 17 | 17 |
| ·691 | 112 | 17 | 6 | ·735 | 120 | 1 | 0 |
| ·692 | 113 | 0 | 12 | ·736 | 120 | 4 | 6 |
| ·693 | 113 | 3 | 19 | ·737 | 120 | 7 | 12 |
| ·694 | 113 | 7 | 1 | ·738 | 120 | 10 | 19 |
| ·695 | 113 | 10 | 8 | ·739 | 120 | 14 | 1 |
| ·696 | 113 | 13 | 14 | ·740 | 120 | 17 | 2 |
| ·697 | 113 | 16 | 20 | ·741 | 121 | 0 | 14 |
| ·698 | 114 | 0 | 3 | ·742 | 121 | 3 | 20 |
| ·699 | 114 | 3 | 9 | ·743 | 121 | 7 | 3 |
| ·700 | 114 | 6 | 16 | ·744 | 121 | 10 | 9 |
| ·701 | 114 | 9 | 22 | ·745 | 121 | 13 | 6 |
| ·702 | 114 | 13 | 4 | ·746 | 121 | 16 | 22 |
| ·703 | 114 | 16 | 12 | ·747 | 122 | 0 | 4 |
| ·704 | 114 | 19 | 17 | ·748 | 122 | 3 | 11 |
| ·705 | 115 | 3 | 0 | ·749 | 122 | 6 | 17 |
| ·706 | 115 | 6 | 6 | ·750 | 122 | 10 | 0 |
| ·707 | 115 | 9 | 12 | ·751 | 122 | 13 | 16 |
| ·708 | 115 | 12 | 19 | ·752 | 122 | 16 | 12 |
| ·709 | 115 | 16 | 1 | ·753 | 122 | 19 | 19 |
| ·710 | 115 | 19 | 8 | ·754 | 123 | 3 | 1 |
| ·711 | 116 | 2 | 14 | ·755 | 123 | 6 | 8 |
| ·712 | 116 | 5 | 20 | ·756 | 123 | 9 | 14 |
| ·713 | 116 | 9 | 3 | ·757 | 123 | 12 | 20 |
| ·714 | 116 | 12 | 9 | ·758 | 123 | 16 | 3 |
| ·715 | 116 | 15 | 16 | ·759 | 123 | 19 | 9 |
| ·716 | 116 | 18 | 22 | ·760 | 124 | 2 | 16 |
| ·717 | 117 | 2 | 4 | ·761 | 124 | 5 | 22 |
| ·718 | 117 | 5 | 11 | ·762 | 124 | 9 | 4 |
| ·719 | 117 | 8 | 17 | ·763 | 124 | 12 | 11 |
| ·720 | 117 | 12 | 0 | ·764 | 124 | 15 | 17 |

| If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | |
|---|---|---|---|
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·765 | 124 | 19 | 0 |
| ·766 | 125 | 2 | 6 |
| ·767 | 125 | 5 | 12 |
| ·768 | 125 | 8 | 19 |
| ·769 | 125 | 12 | 1 |
| ·770 | 125 | 15 | 8 |
| ·771 | 125 | 18 | 14 |
| ·772 | 126 | 1 | 20 |
| ·773 | 126 | 5 | 3 |
| ·774 | 126 | 8 | 9 |
| ·775 | 126 | 11 | 16 |
| ·776 | 126 | 14 | 22 |
| ·777 | 126 | 18 | 4 |
| ·778 | 127 | 1 | 11 |
| ·779 | 127 | 4 | 17 |
| ·780 | 127 | 8 | 0 |
| ·781 | 127 | 11 | 6 |
| ·782 | 127 | 14 | 12 |
| ·783 | 127 | 17 | 19 |
| ·784 | 128 | 1 | 1 |
| ·785 | 128 | 4 | 8 |
| ·786 | 128 | 7 | 14 |
| ·787 | 128 | 10 | 20 |
| ·788 | 128 | 14 | 3 |
| ·789 | 128 | 17 | 9 |
| ·790 | 129 | 0 | 16 |
| ·791 | 129 | 3 | 22 |
| ·792 | 129 | 7 | 4 |
| ·793 | 129 | 10 | 11 |
| ·794 | 129 | 13 | 17 |
| ·795 | 129 | 17 | 0 |
| ·796 | 130 | 0 | 6 |
| ·797 | 130 | 3 | 12 |
| ·798 | 130 | 6 | 19 |
| ·799 | 130 | 10 | 1 |
| ·800 | 130 | 13 | 8 |
| ·801 | 130 | 16 | 14 |
| ·802 | 130 | 19 | 20 |
| ·803 | 131 | 3 | 3 |
| ·804 | 131 | 6 | 9 |
| ·805 | 131 | 9 | 16 |
| ·806 | 131 | 12 | 22 |
| ·807 | 131 | 16 | 4 |
| ·808 | 131 | 19 | 11 |
| ·809 | 132 | 2 | 17 |
| ·810 | 132 | 6 | 0 |
| ·811 | 132 | 9 | 6 |
| ·812 | 132 | 12 | 12 |
| ·813 | 132 | 15 | 19 |
| ·814 | 132 | 19 | 1 |
| ·815 | 133 | 2 | 8 |
| ·816 | 133 | 5 | 14 |
| ·817 | 133 | 8 | 20 |
| ·818 | 133 | 12 | 3 |
| ·819 | 133 | 15 | 9 |
| ·820 | 133 | 18 | 16 |
| ·821 | 134 | 1 | 22 |
| ·822 | 134 | 5 | 4 |
| ·823 | 134 | 8 | 11 |
| ·824 | 134 | 11 | 17 |
| ·825 | 134 | 15 | 0 |
| ·826 | 134 | 18 | 6 |
| ·827 | 135 | 1 | 12 |
| ·828 | 135 | 4 | 19 |
| ·829 | 135 | 8 | 1 |
| ·830 | 135 | 11 | 8 |
| ·831 | 135 | 14 | 14 |
| ·832 | 135 | 11 | 8 |
| ·833 | 136 | 1 | 3 |
| ·834 | 136 | 4 | 9 |
| ·835 | 136 | 7 | 16 |
| ·836 | 136 | 10 | 22 |
| ·837 | 136 | 14 | 4 |
| ·838 | 136 | 17 | 11 |
| ·839 | 137 | 0 | 17 |
| ·840 | 137 | 4 | 0 |
| ·841 | 137 | 7 | 6 |
| ·842 | 137 | 10 | 12 |
| ·843 | 137 | 13 | 19 |
| ·844 | 137 | 17 | 1 |
| ·845 | 138 | 0 | 8 |
| ·846 | 138 | 3 | 14 |
| ·847 | 138 | 6 | 20 |
| ·848 | 138 | 10 | 3 |
| ·849 | 138 | 13 | 19 |
| ·850 | 138 | 16 | 16 |
| ·851 | 138 | 19 | 22 |
| ·852 | 139 | 3 | 4 |

| If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | |
|---|---|---|---|
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·853 | 139 | 6 | 11 |
| ·854 | 139 | 9 | 17 |
| ·855 | 139 | 13 | 0 |
| ·856 | 139 | 16 | 6 |
| ·857 | 139 | 19 | 12 |
| ·858 | 140 | 2 | 19 |
| ·859 | 140 | 6 | 1 |
| ·860 | 140 | 9 | 8 |
| ·861 | 140 | 12 | 14 |
| ·862 | 140 | 15 | 20 |
| ·863 | 140 | 19 | 3 |
| ·864 | 141 | 2 | 9 |
| ·865 | 141 | 5 | 16 |
| ·866 | 141 | 8 | 22 |
| ·867 | 141 | 12 | 4 |
| ·868 | 141 | 15 | 11 |
| ·869 | 141 | 18 | 17 |
| ·870 | 142 | 2 | 0 |
| ·871 | 142 | 5 | 6 |
| ·872 | 142 | 8 | 12 |
| ·873 | 142 | 11 | 19 |
| ·874 | 142 | 15 | 1 |
| ·875 | 142 | 18 | 8 |
| ·876 | 143 | 1 | 14 |
| ·877 | 143 | 4 | 20 |
| ·878 | 143 | 8 | 3 |
| ·879 | 143 | 11 | 9 |
| ·880 | 143 | 14 | 16 |
| ·881 | 143 | 17 | 22 |
| ·882 | 144 | 1 | 4 |
| ·883 | 144 | 4 | 11 |
| ·884 | 144 | 7 | 17 |
| ·885 | 144 | 11 | 0 |
| ·886 | 144 | 14 | 6 |
| ·887 | 144 | 17 | 12 |
| ·888 | 145 | 0 | 19 |
| ·889 | 145 | 4 | 1 |
| ·890 | 145 | 7 | 8 |
| ·891 | 145 | 10 | 14 |
| ·892 | 145 | 13 | 20 |
| ·893 | 145 | 17 | 3 |
| ·894 | 146 | 0 | 9 |
| ·895 | 146 | 3 | 16 |
| ·896 | 146 | 6 | 22 |
| ·897 | 146 | 10 | 4 |
| ·898 | 146 | 13 | 11 |
| ·899 | 146 | 16 | 17 |
| ·900 | 147 | 0 | 0 |
| ·901 | 147 | 3 | 6 |
| ·902 | 147 | 6 | 12 |
| ·903 | 147 | 9 | 19 |
| ·904 | 147 | 13 | 1 |
| ·905 | 147 | 16 | 8 |
| ·906 | 147 | 19 | 14 |
| ·907 | 148 | 2 | 2 |
| ·908 | 148 | 6 | 3 |
| ·909 | 148 | 9 | 9 |
| ·910 | 148 | 12 | 16 |
| ·911 | 148 | 15 | 21 |
| ·912 | 148 | 19 | 4 |
| ·913 | 149 | 2 | 11 |
| ·914 | 149 | 5 | 17 |
| ·915 | 149 | 9 | 0 |
| ·916 | 149 | 12 | 6 |
| ·917 | 149 | 15 | 12 |
| ·918 | 149 | 18 | 19 |
| ·919 | 150 | 2 | 1 |
| ·920 | 150 | 5 | 8 |
| ·921 | 150 | 8 | 14 |
| ·922 | 150 | 11 | 20 |
| ·923 | 150 | 15 | 3 |
| ·924 | 150 | 18 | 9 |
| ·925 | 151 | 1 | 16 |
| ·926 | 151 | 4 | 22 |
| ·927 | 151 | 8 | 4 |
| ·928 | 151 | 11 | 11 |
| ·929 | 151 | 14 | 17 |
| ·930 | 151 | 18 | 0 |
| ·931 | 152 | 1 | 6 |
| ·932 | 152 | 4 | 12 |
| ·933 | 152 | 7 | 19 |
| ·934 | 152 | 11 | 1 |
| ·935 | 152 | 14 | 8 |
| ·936 | 152 | 17 | 14 |
| ·937 | 153 | 0 | 20 |
| ·938 | 153 | 4 | 3 |
| ·939 | 153 | 7 | 9 |
| ·940 | 153 | 10 | 16 |

| If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | | If 200 Grains of Ore give of FINE METAL | One Ton of Ore. will yield of FINE METAL | | |
|---|---|---|---|---|---|---|---|
| *Gr.* | *Oz.* | *Dwts.* | *Grs.* | *Gr.* | *Oz.* | *Dwts.* | *Grs.* |
| ·941 | 153 | 13 | 22 | ·985 | 160 | 17 | 6 |
| ·942 | 153 | 17 | 4 | ·986 | 161 | 0 | 22 |
| ·943 | 154 | 0 | 11 | ·987 | 161 | 4 | 4 |
| ·944 | 154 | 3 | 17 | ·988 | 161 | 7 | 11 |
| ·945 | 154 | 7 | 0 | ·989 | 161 | 10 | 17 |
| ·946 | 154 | 10 | 6 | ·990 | 161 | 14 | 0 |
| ·947 | 154 | 13 | 12 | ·991 | 161 | 17 | 6 |
| ·948 | 154 | 16 | 19 | ·992 | 162 | 0 | 12 |
| ·949 | 155 | 0 | 1 | ·993 | 162 | 3 | 19 |
| ·950 | 155 | 3 | 8 | ·994 | 162 | 7 | 1 |
| ·951 | 155 | 6 | 14 | ·995 | 162 | 10 | 8 |
| ·952 | 155 | 9 | 20 | ·996 | 162 | 13 | 14 |
| ·953 | 155 | 13 | 3 | ·997 | 162 | 16 | 20 |
| ·954 | 155 | 16 | 9 | ·998 | 163 | 0 | 3 |
| ·955 | 155 | 19 | 16 | ·999 | 163 | 3 | 9 |
| ·956 | 156 | 2 | 22 | 1 grain | 163 | 6 | 16 |
| ·957 | 156 | 6 | 4 | 2 | 326 | 13 | 8 |
| ·958 | 156 | 9 | 11 | 3 | 490 | 0 | 0 |
| ·959 | 156 | 12 | 17 | 4 | 653 | 6 | 16 |
| ·960 | 156 | 16 | 0 | 5 | 816 | 13 | 8 |
| ·961 | 156 | 19 | 6 | 6 | 980 | 0 | 0 |
| ·962 | 157 | 2 | 12 | 7 | 1143 | 6 | 16 |
| ·963 | 157 | 5 | 19 | 8 | 1306 | 13 | 8 |
| ·964 | 157 | 9 | 1 | 9 | 1470 | 0 | 0 |
| ·965 | 157 | 12 | 8 | 10 | 1633 | 6 | 16 |
| ·966 | 157 | 15 | 14 | 11 | 1796 | 13 | 8 |
| ·967 | 157 | 18 | 20 | 12 | 1960 | 0 | 0 |
| ·968 | 158 | 2 | 3 | 13 | 2123 | 6 | 16 |
| ·969 | 158 | 5 | 9 | 14 | 2286 | 13 | 8 |
| ·970 | 158 | 8 | 16 | 15 | 2450 | 0 | 0 |
| ·971 | 158 | 11 | 22 | 16 | 2613 | 6 | 16 |
| ·972 | 158 | 15 | 4 | 17 | 2776 | 13 | 8 |
| ·973 | 158 | 18 | 11 | 18 | 2940 | 0 | 0 |
| ·974 | 159 | 1 | 17 | 19 | 3103 | 6 | 16 |
| ·975 | 159 | 5 | 0 | 20 | 3266 | 13 | 8 |
| ·976 | 159 | 8 | 6 | 21 | 3430 | 0 | 0 |
| ·977 | 159 | 11 | 12 | 22 | 3593 | 6 | 16 |
| ·978 | 159 | 14 | 19 | 23 | 3756 | 13 | 8 |
| ·979 | 159 | 18 | 1 | 24 | 3920 | 0 | 0 |
| ·980 | 160 | 1 | 8 | 25 | 4083 | 6 | 16 |
| ·981 | 160 | 4 | 14 | 26 | 4246 | 13 | 8 |
| ·982 | 160 | 7 | 20 | 27 | 4410 | 0 | 0 |
| ·983 | 160 | 10 | 3 | 28 | 4573 | 6 | 16 |
| ·984 | 160 | 14 | 9 | 29 | 4736 | 13 | 8 |

| If 200 Grains of Ore give of | One Ton of Ore will yield of | | | If 200 Grains of Ore give of | One Ton of Ore will yield of | | |
|---|---|---|---|---|---|---|---|
| FINE METAL | FINE METAL | | | FINE METAL | FINE METAL | | |
| *Grs.* | *Oz.* | *Dwts.* | *Grs.* | *Grs.* | *Oz.* | *Dwts.* | *Grs.* |
| 30 | 4900 | 0 | 0 | 74 | 12086 | 13 | 8 |
| 31 | 5063 | 6 | 16 | 75 | 12250 | 0 | 0 |
| 32 | 5226 | 13 | 8 | 76 | 12413 | 6 | 16 |
| 33 | 5390 | 0 | 0 | 77 | 12576 | 13 | 8 |
| 34 | 5553 | 6 | 16 | 78 | 12740 | 0 | 0 |
| 35 | 5716 | 13 | 8 | 79 | 12903 | 6 | 16 |
| 36 | 5880 | 0 | 0 | 80 | 13066 | 13 | 8 |
| 37 | 6043 | 6 | 16 | 81 | 13230 | 0 | 0 |
| 38 | 6206 | 13 | 8 | 82 | 13393 | 6 | 16 |
| 39 | 6370 | 0 | 0 | 83 | 13556 | 13 | 8 |
| 40 | 6533 | 6 | 16 | 84 | 13720 | 0 | 0 |
| 41 | 6696 | 13 | 8 | 85 | 13883 | 6 | 16 |
| 42 | 6860 | 0 | 0 | 86 | 14046 | 13 | 8 |
| 43 | 7023 | 6 | 16 | 87 | 14210 | 0 | 0 |
| 44 | 7186 | 13 | 8 | 88 | 14373 | 6 | 16 |
| 45 | 7350 | 0 | 0 | 89 | 14536 | 13 | 8 |
| 46 | 7513 | 6 | 16 | 90 | 14700 | 0 | 0 |
| 47 | 7676 | 13 | 8 | 91 | 14863 | 6 | 16 |
| 48 | 7840 | 0 | 0 | 92 | 15026 | 13 | 8 |
| 49 | 8003 | 6 | 16 | 93 | 15190 | 0 | 0 |
| 50 | 8166 | 13 | 8 | 94 | 15353 | 6 | 16 |
| 51 | 8330 | 0 | 0 | 95 | 15516 | 13 | 8 |
| 52 | 8493 | 6 | 16 | 96 | 15680 | 0 | 0 |
| 53 | 8656 | 13 | 8 | 97 | 15843 | 6 | 16 |
| 54 | 8820 | 0 | 0 | 98 | 16006 | 13 | 8 |
| 55 | 8983 | 6 | 16 | 99 | 16170 | 0 | 0 |
| 56 | 9146 | 13 | 8 | 100 | 16333 | 6 | 16 |
| 57 | 9310 | 0 | 0 | 101 | 16496 | 13 | 8 |
| 58 | 9473 | 6 | 16 | 102 | 16660 | 0 | 0 |
| 59 | 9636 | 13 | 8 | 103 | 16823 | 6 | 16 |
| 60 | 9800 | 0 | 0 | 104 | 16986 | 13 | 8 |
| 61 | 9963 | 6 | 16 | 105 | 17150 | 0 | 0 |
| 62 | 10126 | 13 | 8 | 106 | 17313 | 6 | 16 |
| 63 | 10290 | 0 | 0 | 107 | 17476 | 13 | 8 |
| 64 | 10453 | 6 | 16 | 108 | 17640 | 0 | 0 |
| 65 | 10616 | 13 | 8 | 109 | 17803 | 6 | 16 |
| 66 | 10780 | 0 | 0 | 110 | 17966 | 13 | 8 |
| 67 | 10943 | 6 | 16 | 111 | 18130 | 0 | 0 |
| 68 | 11106 | 13 | 8 | 112 | 18293 | 6 | 16 |
| 69 | 11270 | 0 | 0 | 113 | 18456 | 13 | 8 |
| 70 | 11433 | 6 | 16 | 114 | 18620 | 0 | 0 |
| 71 | 11596 | 13 | 8 | 115 | 18783 | 6 | 16 |
| 72 | 11760 | 0 | 0 | 116 | 18946 | 13 | 8 |
| 73 | 11923 | 6 | 16 | 117 | 19110 | 0 | 0 |

| If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | | If 200 Grains of Ore give of FINE METAL | One Ton of Ore will yield of FINE METAL | | |
|---|---|---|---|---|---|---|---|
| *Grs.* | *Oz.* | *Dwts.* | *Grs.* | *Grs.* | *Oz.* | *Dwts.* | *Grs.* |
| 118 | 19273 | 6 | 16 | 160 | 26133 | 6 | 16 |
| 119 | 19436 | 13 | 8 | 161 | 26296 | 13 | 8 |
| 120 | 19600 | 0 | 0 | 162 | 26460 | 0 | 0 |
| 121 | 19763 | 6 | 16 | 163 | 26623 | 6 | 16 |
| 122 | 19926 | 13 | 8 | 164 | 26786 | 13 | 8 |
| 123 | 20090 | 0 | 0 | 165 | 26950 | 0 | 0 |
| 124 | 20253 | 6 | 16 | 166 | 27113 | 6 | 16 |
| 125 | 20416 | 13 | 8 | 167 | 27276 | 13 | 8 |
| 126 | 20580 | 0 | 0 | 168 | 27440 | 0 | 0 |
| 127 | 20743 | 6 | 16 | 169 | 27603 | 6 | 16 |
| 128 | 20906 | 13 | 8 | 170 | 27766 | 13 | 8 |
| 129 | 21070 | 0 | 0 | 171 | 27930 | 0 | 0 |
| 130 | 21233 | 6 | 16 | 172 | 28093 | 6 | 16 |
| 131 | 21396 | 13 | 8 | 173 | 28256 | 13 | 8 |
| 132 | 21560 | 0 | 0 | 174 | 28420 | 0 | 0 |
| 133 | 21723 | 6 | 16 | 175 | 28583 | 6 | 16 |
| 134 | 21886 | 13 | 8 | 176 | 28746 | 13 | 8 |
| 135 | 22050 | 0 | 0 | 177 | 28910 | 0 | 0 |
| 136 | 22213 | 6 | 16 | 178 | 29073 | 6 | 16 |
| 137 | 22376 | 13 | 8 | 179 | 29236 | 13 | 8 |
| 138 | 22540 | 0 | 0 | 180 | 29400 | 0 | 0 |
| 139 | 22703 | 6 | 16 | 181 | 29563 | 6 | 16 |
| 140 | 22866 | 13 | 8 | 182 | 29726 | 13 | 8 |
| 141 | 23030 | 0 | 0 | 183 | 29890 | 0 | 0 |
| 142 | 23193 | 6 | 16 | 184 | 30053 | 6 | 16 |
| 143 | 23356 | 13 | 8 | 185 | 30216 | 13 | 8 |
| 144 | 23520 | 0 | 0 | 186 | 30380 | 0 | 0 |
| 145 | 23683 | 6 | 16 | 187 | 30543 | 6 | 16 |
| 146 | 23846 | 13 | 8 | 188 | 30706 | 13 | 8 |
| 147 | 24010 | 0 | 0 | 189 | 30870 | 0 | 0 |
| 148 | 24173 | 6 | 16 | 190 | 31033 | 6 | 16 |
| 149 | 24336 | 13 | 8 | 191 | 31196 | 13 | 8 |
| 150 | 24500 | 0 | 0 | 192 | 31360 | 0 | 0 |
| 151 | 24663 | 6 | 16 | 193 | 31523 | 6 | 16 |
| 152 | 24826 | 13 | 8 | 194 | 31686 | 13 | 8 |
| 153 | 24990 | 0 | 0 | 195 | 31850 | 0 | 0 |
| 154 | 25153 | 6 | 16 | 196 | 32013 | 6 | 16 |
| 155 | 25316 | 13 | 8 | 197 | 32176 | 13 | 8 |
| 156 | 25480 | 0 | 0 | 198 | 32340 | 0 | 0 |
| 157 | 25643 | 6 | 16 | 199 | 32503 | 6 | 16 |
| 158 | 25806 | 13 | 8 | 200 | ( 32666 | 13 | 8 = 20 Cwt. of 112 |
| 159 | 25970 | 0 | 0 | | | | |

[illegible]

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